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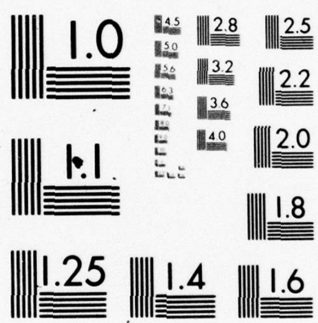

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This program established that fuselage braces (Part No. A51B21683) manufactured by the hot isostatic pressing (HIP) process can satisfy flight requirements for forged parts. The HIP braces withstood 24,000 equivalent flight hours (four design lives) without failure. The projected cost saving related to the utilization of the HIP process in manufacturing of these parts is in the range of 30 to 40%.

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Ti-6Al-6V-2Sn titanium alloys consolidated by HIP exhibited excellent fracture toughness and elongation characteristics, and obtained K_{1c} and percent elongation values in the range of 69-74 ksi $\sqrt{\text{in.}}$ and 15-16%, respectively. Representative F_{tu} and F_{ty} values were approximately 3% below the current design specifications for forgings. However, these values can be increased by minor modifications in the oxygen content within allowable limits. Mechanical properties exhibited isotropic characteristics.

F sub tu

F sub ty

Reproducibility studies indicated that current tight tolerances for machined components may have to be relaxed to assure that all dimensions of hot isostatically pressed (HIP'd) parts meet design specifications.

Experimental welds on HIP and vacuum annealed materials met radiographic and ultrasonic acceptance criteria.

Utilization of the HIP process in manufacturing fatigue-critical airframe components will depend on availability of high-quality powders with certified purity standards to insure freedom from inclusions.

FOREWORD

This Final Report covers the work performed under Contract N00019-76-C-0143 from 1 November 1975 to 31 March 1977. It is published for information only and does not necessarily represent the recommendations, conclusions or approval of the Navy. This work was administered under the technical direction of Mr. W. T. Highberger, Jr. of the Naval Air Systems Command, Washington, D.C. 20360. This program was directed by Mr. Joel Magnuson, Project Engineer, and Mr. Robert Witt, Project Manager, Advanced Materials and Processes Development. Assisting on the program was Mr. David Layton of the Elements and Materials Test Laboratory. Mr. Vijay Chandhok served as the Project Manager for the Crucible Materials Research Center, the major subcontractor on the program.

This report has been reviewed and is approved.

Carl Micillo, Manager
Advanced Materials and
Processes Development

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Section 1

INTRODUCTION

1.1 BACKGROUND

This program, which was a follow-on effort to Navy Contract N00019-74-C-0301, "Manufacturing of Titanium Components by Hot Isostatic Pressing", was designed to verify the flight-worthiness of hot isostatically pressed (HIP'd) airframe components. Many efforts are being directed toward reducing the cost of complex titanium parts, especially those which are manufactured by methods involving extensive machining. The difficulty of forging thin titanium sections, combined with the high cost of machining, has priced titanium out of extensive future aircraft use. For this reason, serious efforts are being expended on new technologies to produce net or near net-shape titanium parts. These include isothermal forging, cold isostatic pressing plus sintering, hot die pressing and hot isostatic pressing (HIP).

Isothermal forging and hot die pressing are limited by part complexity and requirements for elevated-temperature die alloys. Sintering is questionable with respect to the full densification of the component. Hot isostatic pressing (HIP), or the other hand, has been shown to be capable of producing complex parts to full density and does not require the use of high-temperature die alloys.

1.2 PROCESS DESCRIPTION

The HIP process consists of encapsulating metallic powders in a suitably shaped mold, evacuating and sealing the mold assembly, and positioning it in a high temperature/pressure autoclave that contains gaseous media (see Figure 1). As the built-in heaters increase the temperature of the mold material to its softening range, the applied pressure is transmitted to the powder charge through (now flexible) walls of the mold. Particle bonding is further enhanced by diffusion accelerated by high temperature. Titanium alloy powders are especially suitable for processing by HIP because of titanium's ability to dissolve its own (particle surface) oxide at relatively low temperatures; this enables the diffusion process to proceed readily and facilitates bonding.

Since the major factor in machining costs is the number of separate machining steps required, rather than the amount of material removed, net-shape manufacturing represents a realistic approach to reduce the cost of forged titanium parts by 40 percent or more. Other net-shape processes are limited either by obtainable product properties or by part

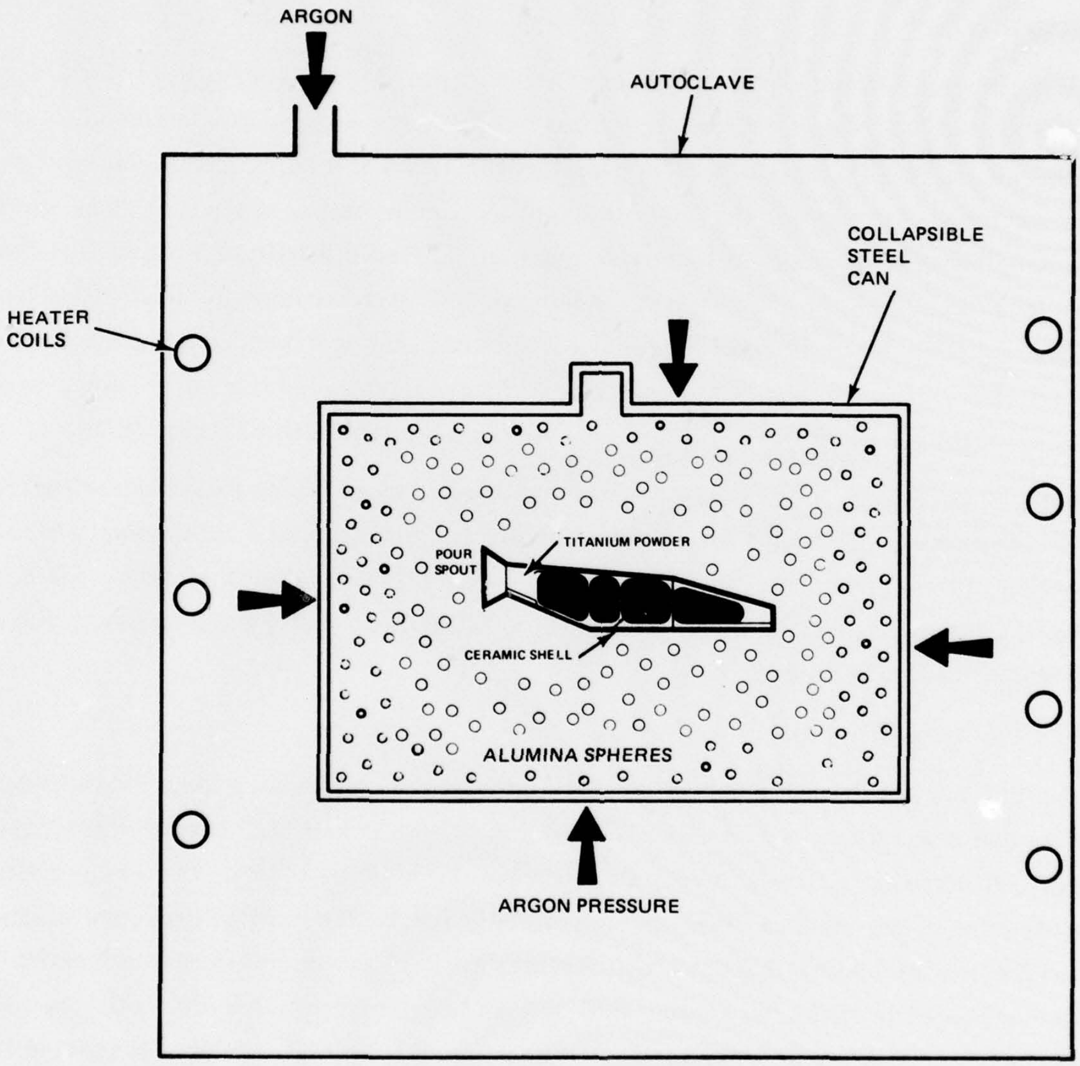

Figure 1. Schematic Presentation of Hot Isostatic Pressing Operation as Performed by Crucible

complexity. The latter is of particular importance, because airframe parts usually consist of deep pockets and thin intersecting webs and ribs to meet high strength-to-weight design requirements. This program, therefore, emphasized the manufacture of a net shape rather than machining preforms. A fuselage brace was selected for this program because it combined typical airframe design in a configuration of convenient size and weight.

1.3 PROGRAM OBJECTIVE

The purpose of this program was to establish the flight-worthiness and reproducibility of an F-14 airframe component manufactured by hot isostatic pressing of pre-alloyed Ti-6Al-6V-2Sn titanium alloy powder.

1.4 PROGRAM APPROACH

The goals of the program were to certify for flight a fuselage brace (Part No. A51B21683) by spectrum fatigue testing to verify its mechanical and physical properties, and to evaluate acceptance criteria for HIP parts.

The program was divided into three tasks (Figure 2). Tasks 1 and 2 were to establish optimum HIP conditions and produce a representative pilot lot for reproducibility and functional tests. Task 3, which ran concurrently with Tasks 1 and 2, was to define nondestructive inspection techniques and standards for the acceptance of HIP components.

HIP processing was performed at the Crucible Materials Research Center (CMR) of Pittsburgh, Pennsylvania. Material processed during the first three HIP runs was to be compared for tensile and fatigue properties and evaluated for stress-relieving requirements. The final parameters for the pilot-lot HIP cycle and the optimized die configuration were to be selected at the conclusion of Task 1.

Task 3 was to establish nondestructive testing techniques for HIP'd Ti-6Al-6V-2Sn titanium alloy powder parts. These studies included hot isostatic pressing of standard blocks with densities in the range of 98-99 percent. These blocks were then to be used to determine the feasibility of detecting density variations by ultrasound velocity measurements. The standards developed in Task 3 were to be applied to the parts produced in the pilot lot in Task 2.

In Task 2, a pilot lot (10 pieces) of the program part (fuselage brace) was to be manufactured for testing to verify flight-worthiness of HIP-produced parts. (Task 2 made use of HIP parameters developed in Task 1 and the NDI criteria developed in Task 3.) The fuselage brace was to be subjected to spectrum fatigue loading of up to four times the aircraft design life. In addition, bulk HIP'd Ti-6Al-6V-2Sn blocks were to be machined into test specimens

for generation of mechanical property data, including tensile, fatigue and fracture toughness. In addition, net-shape fatigue specimens (Appendix B) were to be manufactured for fatigue evaluation of as-HIP surfaces.

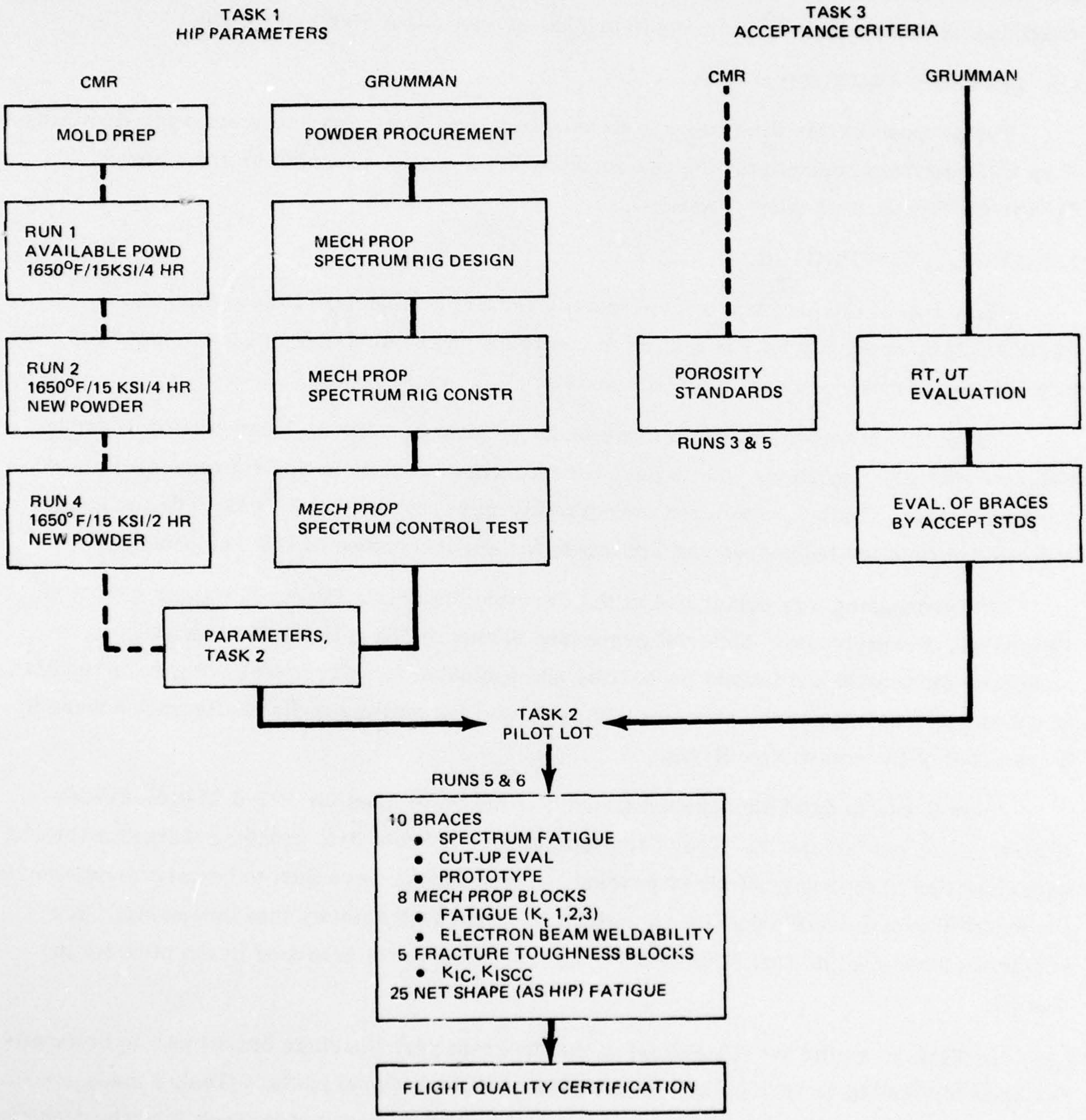

Figure 2. Program Flow Chart

Section 2

PARAMETER SELECTION

2.1 INTRODUCTION

Based on Phase 1 results (Reference 1), it was concluded that mechanical properties of hot isostatically pressed configurations depend on time at pressure and temperature, and oxygen level of the material. Task 1 of the present Phase 2 effort was designed to evaluate effects of increased time at temperature and pressure on mechanical properties and to improve dimensional tolerances of the selected part. Special efforts were required, for instance, to eliminate beveling at the lower base of the brace.

In considering the effects of HIP parameters, it is essential to realize that temperature/pressure profiles on heating and cooling form an integral part of the HIP cycle. As shown in Figure 3, soaking at the HIP temperature (1650°F) for 8 hours is required to bring the heavily insulated mold assembly (Figure 1) to the required HIP temperature; therefore, the true cycle begins 10 hours after the starting time. Pressure is applied after the autoclave has been at temperature for 4 hours, as shown by the dotted line in Figure 3.

The powder utilized in the Phase 2 experimental studies was produced by REP techniques, identical to those used in Phase 1. Powder remaining from Phase 1 was utilized in the first HIP run to expedite the refinement of dimensional tolerances.

2.2 OBJECTIVE

The objective of Task 1 was to extend the parametric results of Phase 1 to improve tensile strength of Ti-6Al-6V-2Sn configurations manufactured by HIP without sacrificing the excellent ductility and toughness already achieved, and to concurrently refine dimensions and contours of the mold to improve dimensional tolerances of HIP'd parts.

2.3 PROCEDURE

The powder used for this program was manufactured by Nuclear Metals by means of their Rotating Electrode Process (REP). Table I gives the chemistry of the two powder lots used. Figure 4 gives their respective mesh distributions. It can be seen that the particle size for the new powder lot was shifted somewhat toward finer mesh sizes. The Ti-6Al-6V-2Sn configurations utilized for this program were made by the Crucible Materials Research Center (CMR) of Pittsburgh, Pennsylvania, using their patented ceramic mold

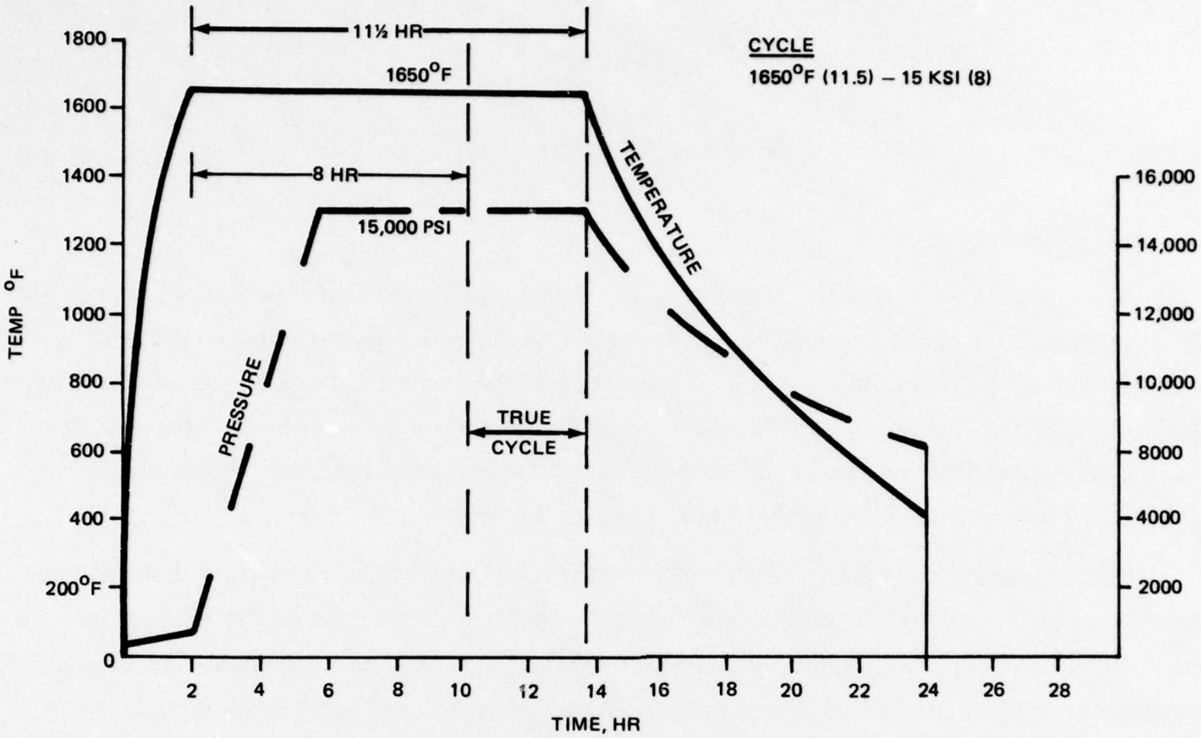

Figure 3. Typical HIP Cycle

Table I. Chemical Analysis of REP Powder

POWDER	ELEMENT WT, %									
	A1	V	Sn	Fe	O	Cu	C	N	H	Ti
RUN 1	5.6	5.4	1.8	0.62	0.19	0.48	0.04	0.01	0.001	Balance
RUNS 2-6	5.7	5.5	2.0	0.74	0.18	0.69	0.02	0.01	0.009	Balance

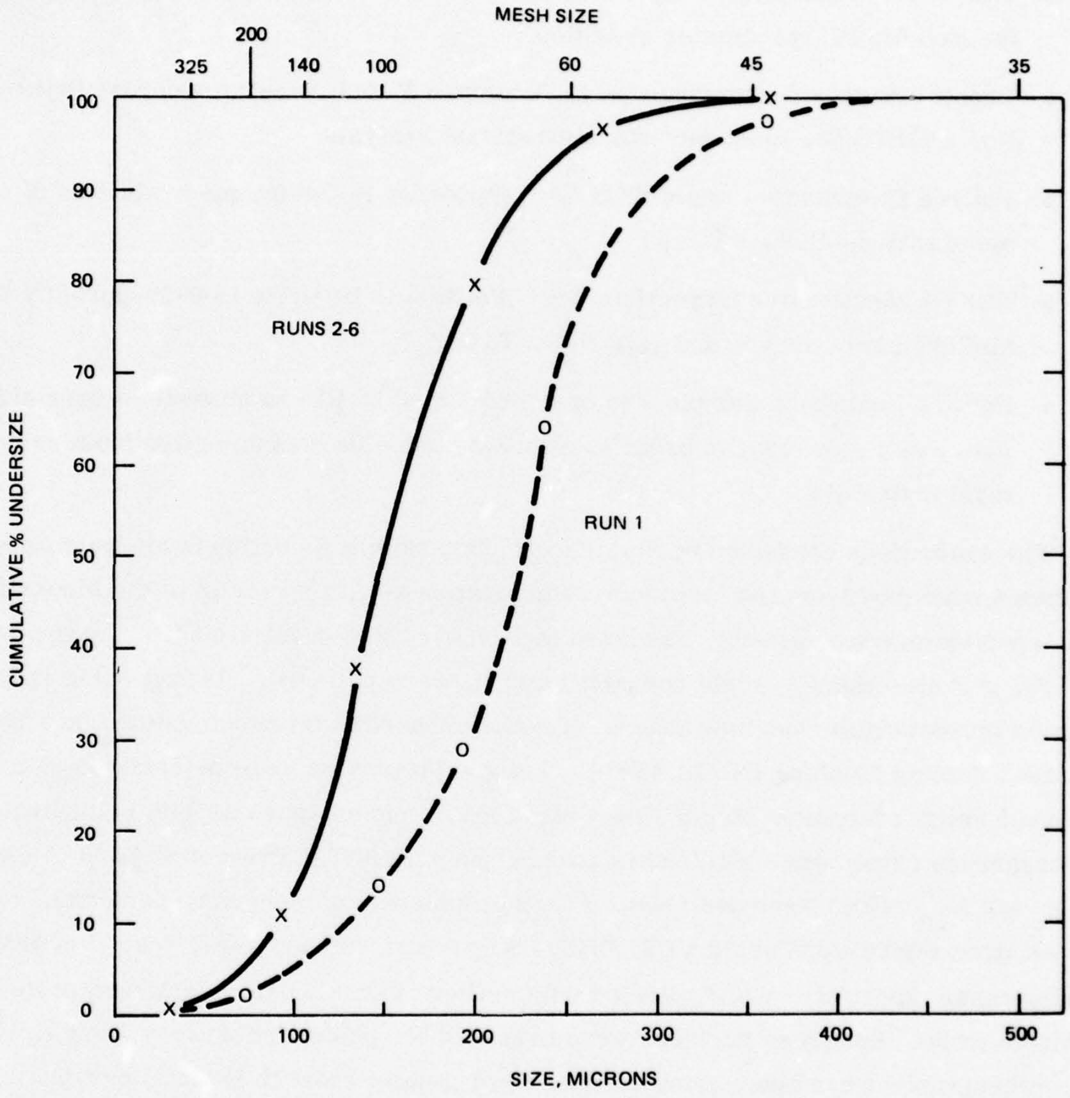

Figure 4. Particle Size Distribution of Ti-6Al-6V-2Sn Titanium Alloy Powder

system. The cycles investigated during the Task 1 study are shown in Table II. The configurations HIP'd in each run are listed in Table III. These configurations were designed for the following purposes:

- Test Block - for 4-in. fatigue coupons (Appendix B) and other test configurations to be used for HIP parameter selection
- Brace Fitting - the program part, Grumman F-14A Fuselage Support Brace, Part No. A51B21683, to be used for dimensional analysis
- Fatigue Specimen - Coupon TGS 5771 (Appendix B) for fatigue evaluation of specimens with as-HIP surfaces
- NDI (Nondestructive Inspection) Test Blocks - to be HIP'd to 0-2% porosity for establishment of acceptance criteria in Task 3
- Powder Evaluation Sample - to be consolidated by HIP in absence of ceramic molds (i.e., in a steel can) in order to eliminate possible contamination from ceramic mold materials.

The evaluations conducted during the HIP Parameters Selection Study were based on specimens machined from the test block configurations. After receipt of the blocks from CMR, specimens were saw-cut, machined and vacuum stress relieved (10^{-5} microns Hg at $1300^{\circ}F$). All specimens, except compact tension, were polished. Radiographic inspection was used on rectangular configurations. Tensile properties were determined on a Riehle Universal Testing Machine (Model SF1U). Fatigue properties were determined on a Sonntag Universal Fatigue Machine (Model FH60) using axial tension-tension (R=0.1) loading. Fracture-toughness values were obtained in accordance with ASTM Procedure E399 (Appendix B); both K_q and K_{1c} values were evaluated. Fractographic evaluations were performed by scanning-electron microscope using a Cambridge Stereoscan 600 and x-ray spectrum analysis. Metallographic specimens were polished using silicon carbide paper, diamond paste and aluminum oxide. Specimen surfaces were prepared for photomicrography using Keller's etch; micrographs were taken using a Bausch and Lomb Research II Metallograph.

2.4 RESULTS

2.4.1 Tensile Tests

Representative tensile test data generated in Task 1 are listed in Table IV together with the minimum requirements of Grumman Specification GM 3117 for forgings. It is evident that tensile properties of HIP'd Ti-6Al-6V-2Sn titanium alloy depend greatly on the

Table II. HIP Matrix for Task 1 — Present Investigation

RUN NO.	TRUE CYCLE DURATION (i.e., MINUS FIRST 8 HOURS OF TEMP SOAK AT 1650°F)	OXYGEN CONTENT, % WT
PREVIOUS EFFORT	2	0.19
1	4	0.19
2	4	0.18
3	3	0.18
4	2	0.18

Table III. Parts HIP'd Under Present Program

HIP RUN	COMPACT NO.	POWDER LOT	MESH SIZE	PART DESCRIPTION	HIP RUN
1	SM-402	A304039	-35	Test Block 5-½" x 4-½" x 1-¼"	KBI 287A
	SM-403			Test Block 5-½" x 4-½" x 1-¼"	
	SM-404			Brace Fitting A51B21683	
	SM-405			Brace Fitting A51B21683	
	SM-406			Powder Evaluation Sample 3" rd x 8"	
2	SM-432	NB 4158	-35	Test Block 5-½" x 4-½" x 1-¼"	KBI 300A
	SM-430B			Fatigue Specimen TGS5771	
	SM-431A			Two NDI Test Blocks	
	SM-430A			Fatigue Specimen TGS5771	
3	SM-446	NB 4158	-35	Brace Fitting A51B21683	CMR 65
	SM-447			Brace Fitting A51B21683 (Vacuum Annealed - 1300 F 2 hr)	
	SM-450A			Two NDI Test Blocks	
	SM-450B			Two NDI Test Blocks	
	SM-449A & B			Two NDI Test Blocks	
4	SM-485	NB 4158	-35	Test Block 5-½" x 4-½" x 1-¼"	CMR 78
	SM-487			Brace Fitting A51B21683 (Vacuum Annealed - 1300 F 2 hr)	
	SM-488			Brace Fitting A51B21683 (Vacuum Annealed - 1300 F 24 hr)	

Table IV. Effect of Annealing on Strength and Ductility of HIP'd Ti-6Al-6V-2Sn Titanium Alloy Powder

AS-MACHINED HIP'd PROPERTIES			HIP'd PROPERTIES AFTER ANNEALING AT 1300°F			
POWDER LOT	304039	4158	304039	4158	4158	Grumman
PPM O_2	1900	1800	1900	1800	1800	Forging Specification
HRS AT 1300°F	None	None	2 Hr	24 Hr	24 Hr	GM 3117
HIP CYCLE	Run 1	Run 2	Run 1	Run 2	Run 4	
F_{tu}, HR	149.1	144.7	148.0	142.5	144.2	150.0
F_{ty}, HR	142.2	134.6	141.0	135.1	136.4	140.0
ELONG, %	17.7	14.7	18.0	22.0	20.0	8.0
RA, %	35.1	43.9	43.0	47.8	42.2	20.0

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oxygen content of the original powder. For powder lot No. 304039 used in Phase 1 investigations and the first HIP run of Task 1 (Phase 2), a 100-ppm increase in the oxygen content over corresponding value of Lot 4158 resulted in a 4-5 ksi increase in F_{tu} and more than a 7 ksi increase in F_{ty}. F_{ty} and elongation of configurations HIP'd from Phase 1 powders exceeded minimum requirements of Grumman Specification GM 3117; elongation values obtained exceeded minimum requirements by a factor of 2. Although F_{tu} values of shapes consolidated from this powder were slightly below (less than one ksi) the minimum requirements, a 50 ppm increase in the oxygen level could definitely bring F_{ty} into the required range.

Annealing at 1300°F for 2 hours did not appreciably affect mechanical properties; annealing for 24 hours at 1300°F, on the other hand, resulted in approximately a 30% increase in elongation without detrimental effects on ultimate and yield tensile strengths.

2.4.2 Effects Of Isotropy

Although tensile properties of HIP-consolidated configurations may prove to be somewhat lower than those taken in the longitudinal direction of forgings (Table V), isotropy of HIP micro-structure and properties may be a definite asset from a design standpoint. Excellent fracture toughness of HIP configurations, which is far superior to that of the wrought counterparts, represents another advantage of HIP technology. In the present study, isotropy of microstructure and mechanical properties were established by metallography and comparable test data for randomly selected test specimens, respectively.

2.4.3 Fatigue Endurance

Fatigue test data on Ti-6Al-6V-2Sn titanium alloy specimens (Configuration 45 MPS 196, Appendix B), consolidated by HIP in the course of Runs 1 through 4, are presented in Figure 5. The legend indicates the serial numbers of HIP runs and the annealing heat treatment parameters utilized. These data indicate that all but two of the data points fall within the typical data band for annealed forgings. One of these two specimens, identified by a subscript "M", was subjected to a fractographic evaluation, which established that the failure initiated at a ceramic inclusion as detailed in Subsection 2.4.4.

Vacuum annealing at 1300°F did not have detrimental effects on fatigue endurance. There were no appreciable variations in fatigue endurance of materials processed in the course of 2-hour and 4-hour HIP runs.

Table V. Tensile Properties of Annealed Forgings and HIP'd Specimen

SOURCE (OXYGEN)	F_{tu}, KSI	F_{ty}, KSI	ELONG, %	RA, %
TMCA (Ref 4) (0.10)	147	137	20	34
TMCA (Ref 4) (0.16)	150	140	20	38
MARTIN MARIETTA (Ref 5)	155	149	16	43
GRUMMAN (Ref 6)	151	144	18	44
BOEING-N.AMERICAN(Ref 7) (0.15)	148	142	11	44
NASA LEWIS (Ref 5) (0.12)	148	140	16	28
GRUMMAN (Ref 9) (0.15)	160	152	15	–
a ANNEALED FORGING AVERAGE	151.2	143.4	16.5	38.5
b GRUMMAN SPECIFICATION	150	140	8	20
c HIP RUN 1 (2-HR ANNEAL) (0.19)	148	141	18	43
d % DIFFERENCE FROM (a)	–2.1	–1.7	+9.1	+11.7
e % DIFFERENCE FROM (b)	–1.3	+0.7	+125	+115

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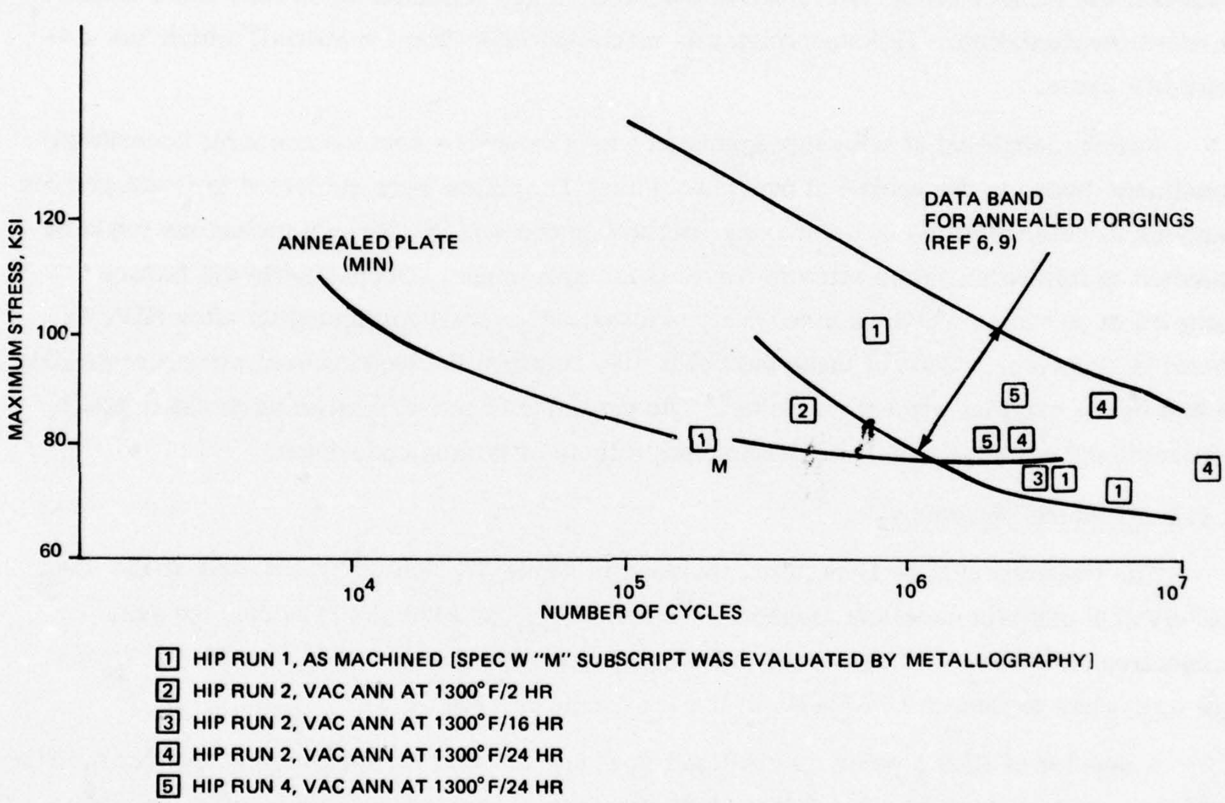


Figure 5. Fatigue Endurance of Ti-6Al-6V-2Sn Titanium Alloy Powder Consolidated by HIP at 1650° F

2.4.4 Fractographic Analysis

Figure 6a shows a failure initiation site with the beach marks from the origin. Figure 6b shows a particle at the fracture initiation site, visible in Figure 6c as a teardrop shape. An x-ray scan revealed the presence of aluminum and silicon (Figures 6c and d) or their compounds (since detection by the x-ray technique is limited to the heavier elements). Note that the cleaved grains surrounding the particle are equiaxed alpha and, thus, have no particular orientation. This specimen was machined from Run 1 material, which has a 4-hour HIP cycle.

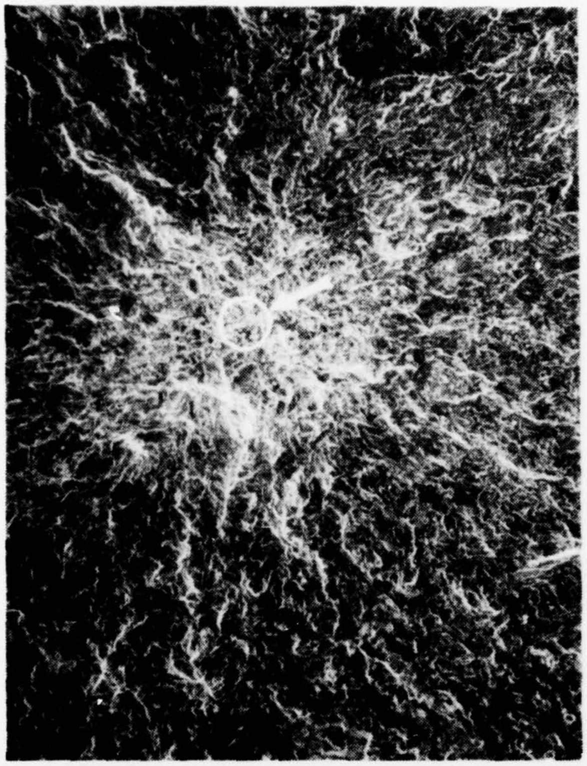
Failure initiation at a foreign inclusion was a cause for serious concern; accordingly, specimens tested in the course of previous (Phase 1) studies were subjected to fractographic analysis to determine the mode of their fracture initiation. No foreign inclusions could be detected at failure initiation sites in any of these specimens. Occasionally the failure initiated at particles which retained their original (spherical) configuration after HIP, as shown in Figure 7. Most of these particles also retained the basketweave structure similar to that of the original powder particles. The presence of non-deformed particles ("ghost spheres") did not appear to have detrimental effects of fatigue endurance.

2.4.5 Fracture Toughness

The previous (Phase 1) results, included in Table VI, demonstrated that HIP'd Ti-6Al-6V-2Sn exhibits excellent toughness (70 and 60 K_{1c} at 1400 and 1900 ppm oxygen, respectively). These values exceed the normal range for Ti-6Al-6V-2Sn of 35-60 K_{1c} and are equivalent to that of Ti-6Al-4V in the RA condition (Figure 8).

A new lot of REP powder was utilized in Phase 2. One K_q specimen (Appendix A, TGS 109345) machined from the Run 2 test block and tested, yielded a K_q value of 85 ksi/inch, which is very high for this level of oxygen (1800 ppm). This test showed that the material exceeds by far the minimum Grumman requirement of 50 K_{1c} for critical components.

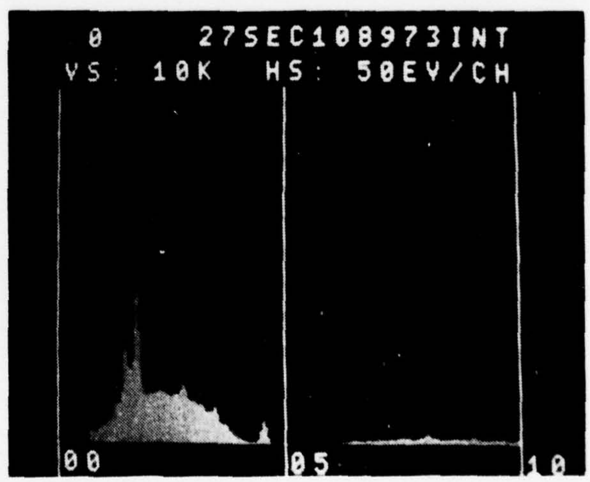
A scanning electron microscopic analysis of the fracture surface of a HIP compact tension fracture specimen reveals the topography to be different from that of wrought material (Figure 9). Contrasting with the smooth flow lines of the control specimen, the HIP fracture surface is pebbly and lacks distinct flow lines. The pebbly surface is the result of the exposure of many spherical grains in relief (Figure 10) by the diversion of the crack tip at prior particle boundaries. This diverted crack propagation apparently explains the high



A. FRACTURE INITIATION AREA (100X MAG)

B. PARTICLE AT FRACTURE INITIATION SITE (1500X MAG)

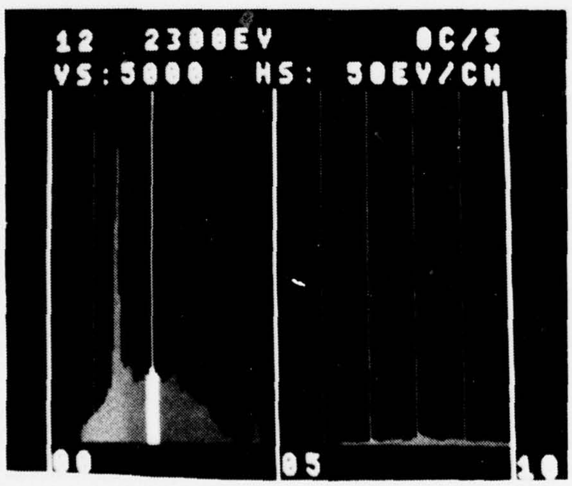
C. X-RAY SCAN



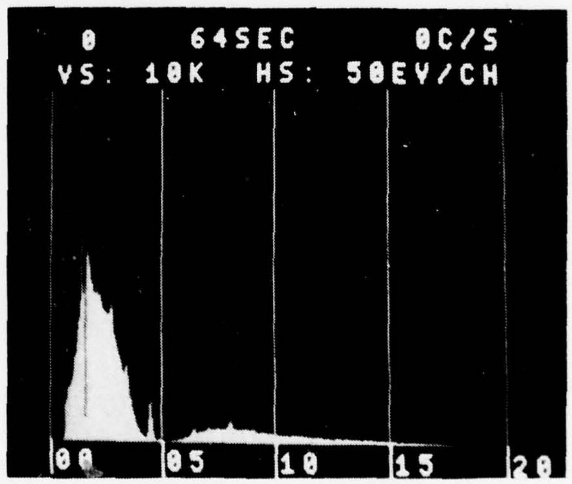
D. X-RAY PLOT

Figure 6. Scanning-Electron-Microscope Examination of Specimen No. 5 (Run 1)

A. FRACTURE INITIATION SITE (400X MAG)



B. X-RAY SCAN OF INITIATION SITE (400X MAG)



C. X-RAY SCAN OF MATRIX (400X MAG)

Figure 7. Analysis of Specimen from Phase 1

Table VI. Effect of Cycle Duration and Oxygen Content on HIP'd Ti-6Al-6V-2 Sn Titanium Alloy Powder

POWDER	OXYGEN CONTENT	TOUGHNESS (Kq)	FRACTURE TOUGHNESS K_{1c}, Ksi $\sqrt{IN.}$	CYCLE LENGTH, HR
PHASE 1	1400	68.9	70.7	2
PHASE 1	1900	–	60.5	2
PHASE 2	1800	85.0	–	4

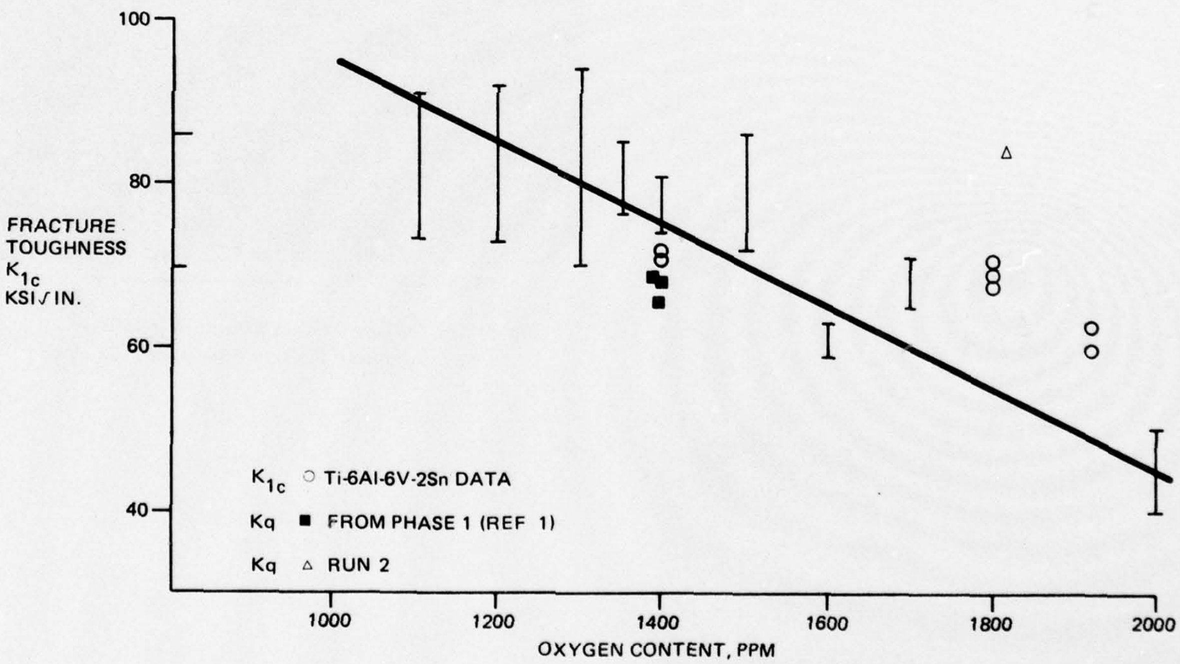


Figure 8. Fracture Toughness of Recrystallized Annealed Ti-6Al-4V (Ref 3) Compared to HIP'd Ti-6Al-6V-2Sn

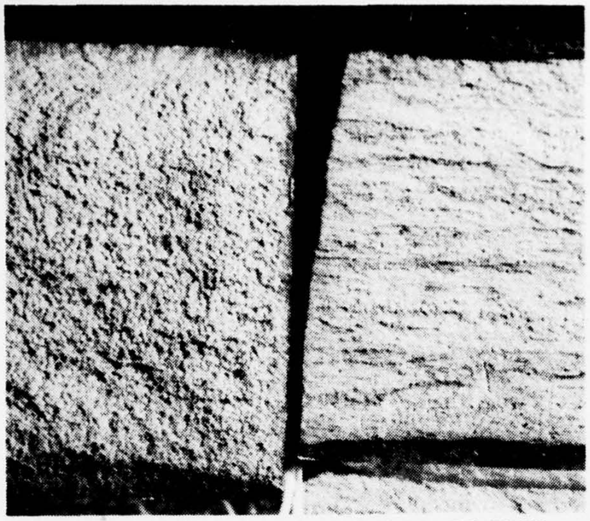


Figure 9. Fractographic Analysis (5x Mag) of K_{1c} Specimen

Figure 10. Spherical Grain Structure in HIP'd Compact Tension Specimen (10x Mag)

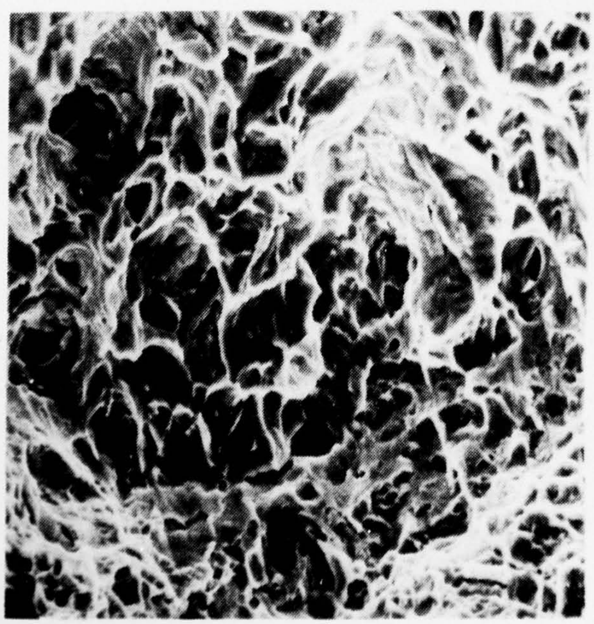


Figure 11. Ductile Rupture of HIP'd Prior-Particle-Spherical Grain (800x Mag)

degree of toughness. At greater magnification (Figure 11), two of these spherical indications can be seen. They are characterized by a ductile rupture with no indication of brittleness within the grains or at their boundaries.

2.4.6 Microstructure

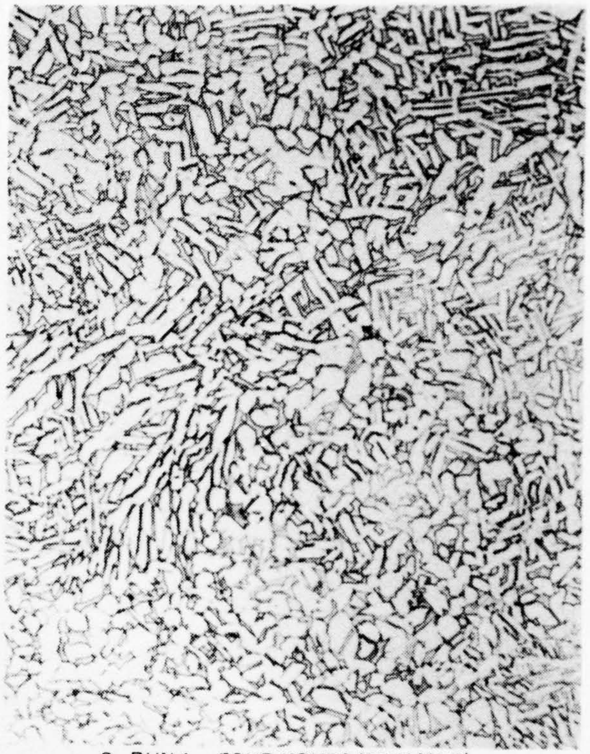
Figure 12 shows that there are virtually no discernible microstructural differences among specimen subjected to 2- and 4-hour HIP cycles. Isolated occurrences of undeformed "ghost" particles referred to in Subsection 2.4.2 were detected throughout the matrix. The basket-weave microstructure of these particles suggests that the transformation of the original dendritic structure to equiaxed alpha-beta structure has been retarded by the lack of deformation of these particles in the course of HIP processing.

2.4.7 Evaluation of Net Shapes-Dimensional Tolerances of HIP Braces

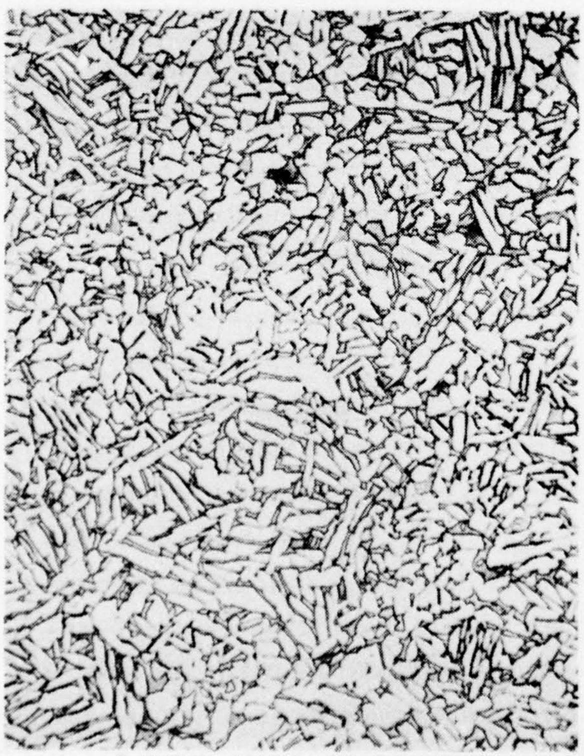
In considering advantages of hot isostatic pressing (HIP), it is essential to realize, that in order to maximize the cost-effectiveness of the process, the parts will have to be produced to net (final) configurations. Even if only superficial machining is required, the cost advantages are substantially reduced due to the setup costs of machining operations. Accordingly, it is essential to refine the process to bring the maximum number of dimensions within the design tolerances or, whenever possible, to consider relaxation of design specifications. Since, prior to the HIP operation, the density of the powder charge is only about 65 percent of that of the fully densified material (thus, considerable volume changes occur on densification), it was anticipated that it may prove to be difficult to meet flatness requirements for mating surfaces and machining would be required to accomplish this objective.

The approach normally used in refining configurations of HIP components consists of machining the die for the wax pattern (to be subsequently used in manufacturing of shell molds for HIP) to undersize dimensions and progressively enlarging the die as required by results of dimensional analyses on HIP parts. Figure 13 shows target dimensions for the selected part (A51B21683). Appendix C lists all actual measurements obtained in the course of dimensional analyses. Mating surfaces for which machining appeared to be mandatory are marked in Figure 13 with the symbol " $\overset{m}{\surd}$. " Table VII lists critical target dimensions and variations from these dimensions exhibited by configurations manufactured in the course of consecutive HIP runs in Phases 1 and 2. The data presented indicate that all but one of final (Run 4) dimensions are well within the limits of existing drawing specifications. Dimension No. 28 was only 0.005 inch below the specified minimum.

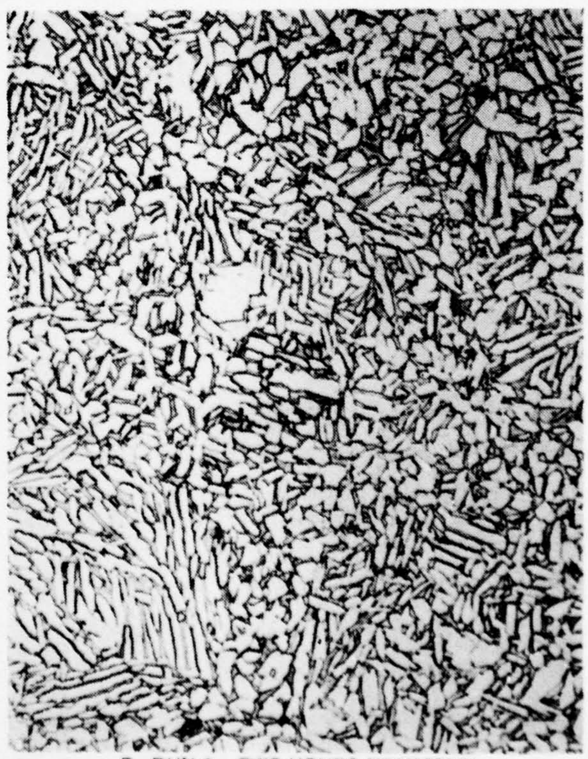
A. RUN 1 – FOUR HOURS (100X MAG)



B. RUN 1 – FOUR HOURS (250X MAG)



C. RUN 2 – FOUR HOURS (250X MAG)



D. RUN 4 – TWO HOURS (250X MAG)

Figure 12. Effect of HIP Cycle Duration on Microstructure of Ti-6Al-6V-2Sn Titanium Alloy

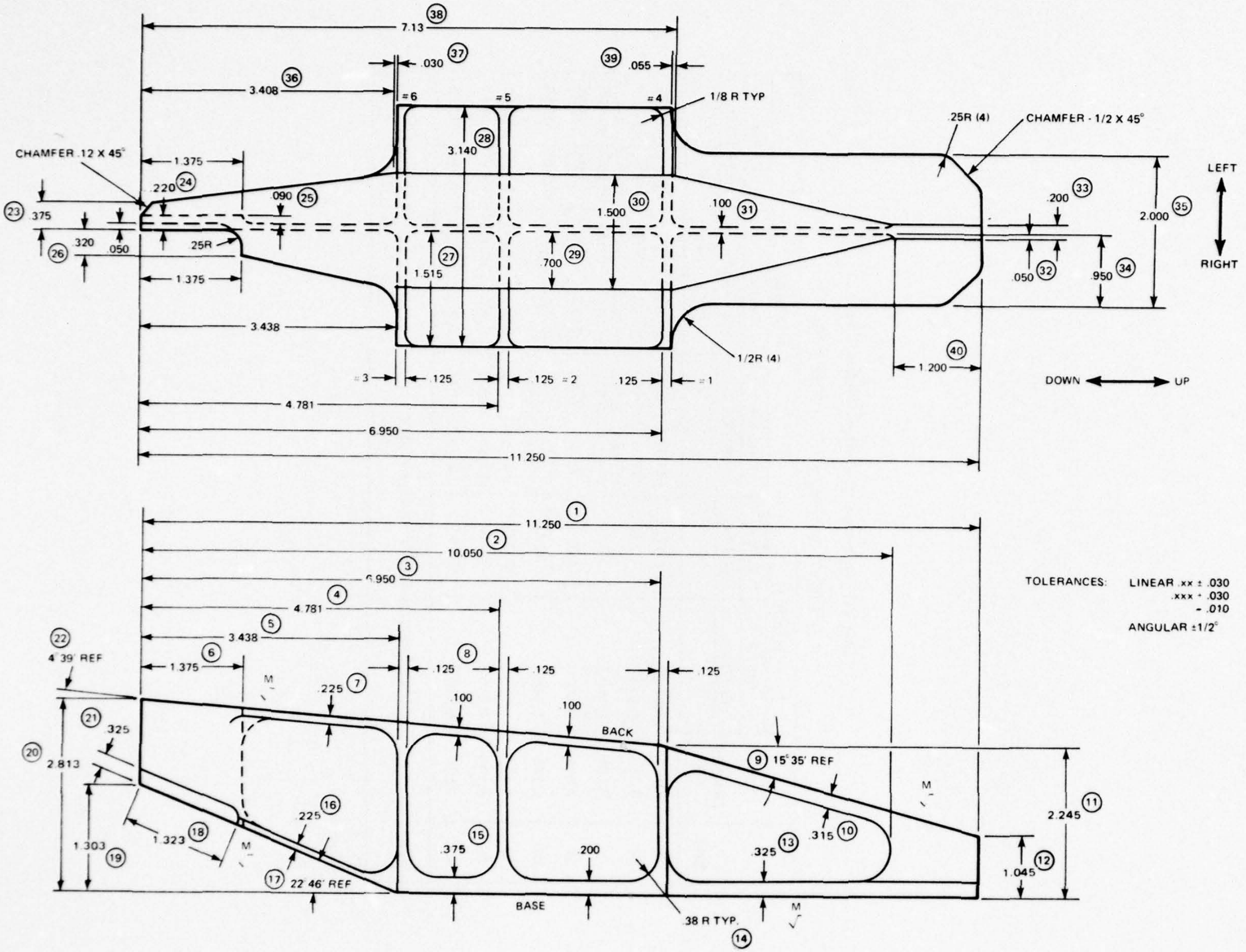


Figure 13. Target Dimensions for Part No. A51B21683

Table VII. Dimensional Control (Phases 1 & 2)

DEVIATIONS FROM TARGET DIMENSIONS

DIM.	TARGET (T)	PHASE 1			PHASE 2					
		RUN 2		RUN 3	RUN 1		RUN 3		RUN 4	
		PART NO.		PART NO.	PART NO.		PART NO.		PART NO.	
		1		1	1	2	1	2	1	2
4	4.781	+0.011		- 0.012	+0.007	+0.005	+0.022	+0.007	+0.007	+0.002
5	3.438	+0.028		0.000	0.000	0.000	0.000	0.000	0.000	0.000
8	0.125	- 0.010		0.000	+0.009 +0.012	+0.009 +0.012	+0.003 +0.014	+0.008 +0.020	- 0.007 +0.006	- 0.005 +0.005
27	1.515	- 0.025		+0.035	- 0.030	- 0.030	- 0.025	- 0.015	+0.003	+0.003
28	3.140	- 0.060		+0.040	- 0.055 - 0.025	- 0.055 - 0.025	- 0.060	- 0.055	- 0.015	- 0.015
30	1.500	- 0.105		+0.066	- 0.010	- 0.010	- 0.040	- 0.035	- 0.010	- 0.005
31	0.100	- 0.010		- 0.010	+0.020	+0.020	0.000	- 0.006	- 0.010	- 0.010
35	2.000	- 0.030		+0.067	- 0.015	- 0.015	- 0.025	- 0.025 - 0.005	+0.010	+0.015

PEMISSABLE TOLERANCES PER DRAWING: .XXX = +0.030
- 0.010

NOTE:

2.4.8 HIP Processing

Figure 14 shows a 60-inch-diameter autoclave utilized in HIP processing. Figure 15 illustrates typical steps in manufacturing titanium components by HIP.

2.4.9 Metallography of Net Shapes Produced by HIP

Metallographic spotchecks on braces manufactured in the course of Runs 1 and 2 (Phase 2) indicated that the microstructures varied considerably from that of more massive rectangular blocks processed during identical HIP runs. Figure 16 illustrates the typical microstructure detected in braces. This microstructure indicated that the β-transus temperature for the brace material may have been exceeded in the course of HIP processing. Since temperature charts for these autoclave runs failed to show any anomalies, an investigation was conducted to detect other possible variations in processing parameters. This study revealed that a new batch of spherical ceramic powder was used by CMRC in these HIP runs and some pickup may have occurred from moisture in inadequately outgassed ceramic. Available data indicate that the β-transus temperature for Ti-6Al-6V-2Sn titanium alloy is depressed 7-10^{o}F per 100 ppm of absorbed hydrogen. The proposed hypothesis was supported by the fact that signs of β-transformation were detected only in braces, i.e., in relatively thin sections of processed shapes. Chemical analyses of specimens removed from braces processed in these runs indicated the hydrogen content was in the range of 1480 to 1690 ppm in contrast to corresponding values in the range of 307 to 478 ppm obtained for massive rectangular blocks. Run No. 3 was designed to manufacture an additional brace configuration using adequately outgassed ceramic materials to ascertain that the problem had been eliminated. The hydrogen content of this brace was determined to be 332 ppm.

Subsequent studies revealed that the excessively high hydrogen contents can be reduced to 188 ppm by vacuum annealing at 1300^{o}F. This treatment also restored the normal α-β microstructure as shown in Figure 17. Results of all hydrogen analyses conducted in the course of these studies are summarized in Table VIII.

2.4.10 Specimens Designed for Fatigue Tests on As-HIP Surfaces

The configuration shown in Appendix B (TGS 5771) was designed to yield fatigue specimens with oversized grip sections (for subsequent machining) and net dimensions of the reduced test sections to generate data on fatigue endurance of shapes with as-HIP surfaces.

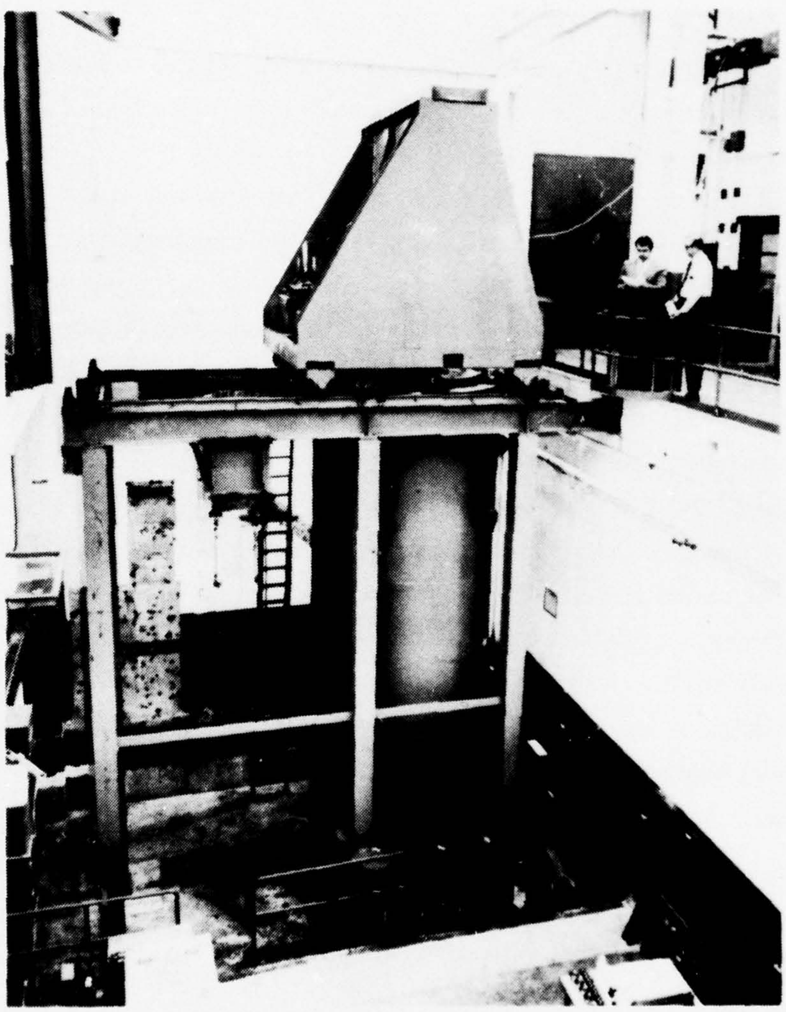


Figure 14. 60-Inch Inside-Diameter Battelle Autoclave

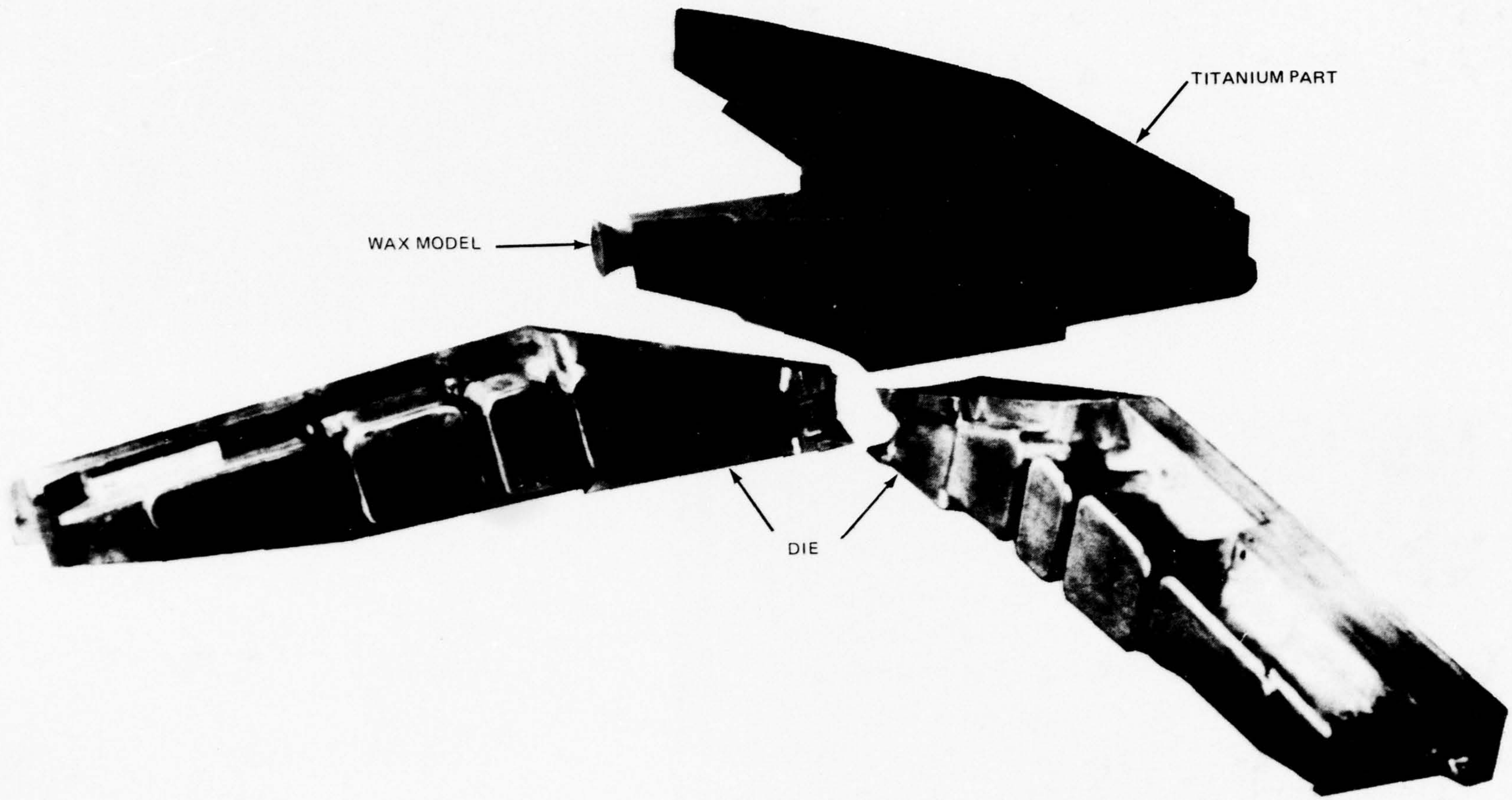


Figure 15. Steps in Production of HIP'd Titanium Component

Figure 16. Beta-Transformed Structure in Braces Manufactured in Runs 1 and 2 (250x Mag)

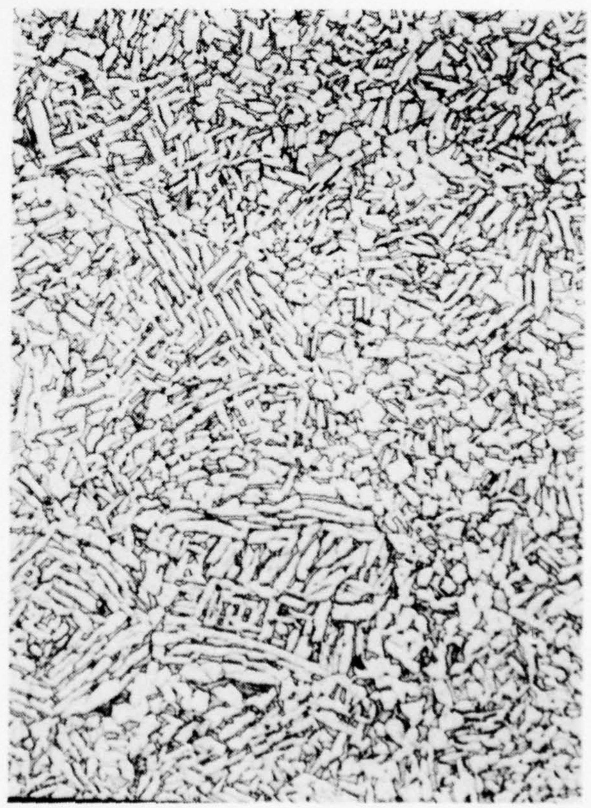


Figure 17. Microstructure of Part No. A51B21683 After Vacuum Annealing (250x Mag)

Table VIII Hydrogen Analyses Performed During Phase 2

POWDER LOT	*NM QCL HYDROGEN, PPM	SPECIMEN	HEAT TREATMENT	LAB	HYDROGEN, PPM	HIP RUN
304039,		Test Coupon	None	GAC	277	
PHASE 1	15	Brace (Piece Cut up)	1300°F Vac Ann	GAC	188	
		Test Coupon	1300°F Vac Ann	GAC	60	1
		2" Block	None	CMR	315	
		2" Block	None	CMR	307	
		Brace	None	CMR	1480	
		Brace	None	CMR	1690	
		Steel Cylinder	None	CMR	68	
4158	90	Test Coupon	1300°F Vac Ann	GAC	175	2
		Test Coupon	Air Furn Ann	GAC	175, 164	5
PHASE 2		Test Coupon	Air Furn Ann	GAC	170, 157	5
		NDI Std	None	CMR	332	3
		Brace	None	CMR	478	4
		Brace	None	CMR	473	4

*Nuclear Metals Quality Control Lab

Significant differences in the masses of grip and test sections make it difficult to maintain integrity of the shell molds in the course of processing. Damaged molds caused external and internal imperfections that rendered specimens unsuitable for testing. Wall thickness of ceramic molds was increased for the subsequent lot of specimens that were manufactured in Run 5.

2.4.11 Selection of Parameters for Reproducibility (Pilot) Lot

Results of evaluations conducted in the course of Task 1 suggested that both 2- and 4-hour cycles are suitable for HIP processing of selected parts. Crucible indicated, however, that selection of 4-hour cycles for production-size lots will unfavorably affect the cost-effective utilization of the autoclave. Accordingly, a compromise was reached and a 3-hour cycle at 1650°F and 15 ksi was selected for the pilot lot manufactured in Task 2.

Section 3

FLIGHT QUALIFICATION TESTS AND REPRODUCIBILITY STUDIES

3.1 INTRODUCTION

After development of the required HIP parameters had been completed in Task 1, Task 2 evaluations could be initiated. These studies were designed to verify the flight-worthiness of HIP'd braces (Part No. A51B21683) under simulated flight conditions, to obtain additional data on mechanical properties (particularly fracture toughness characteristics) and to check on the reproducibility of dimensions obtainable in the course of a pilot lot.

3.2 FUNCTIONAL QUALIFICATION SPECTRUM TEST

3.2.1 Design of the Test

To verify the flight-worthiness of the HIP braces, a truncated 12-level spectrum fatigue test was designed based on the actual 212-level load spectrum experienced by the wing box in flight. Figure 18 shows that, for the identical KN factor, the truncated spectrum selected is more severe than the actual spectrum. The acceptance criteria was based on four life cycles, i.e., 24,000 equivalent flight hours (EFH).

The test assembly designed was an actual replica of a 20-in.-long section of the wing center-section as shown in Figures 19 and 20. The test braces were shot peened to 0.1 Almen intensity level and installed in the test assembly as shown in Figure 21. The loads were introduced through the outside fuselage attachment fitting (Part No. A51B21518) as shown in Figure 19. The completed test assembly shown in Figure 22 simulated accurately the actual stiffness of the wing center-section in the location of braces. Strain gages were positioned on the test braces in locations of maximum anticipated stresses as shown in Figure 20.

3.2.2 Test Procedure and Results

A spectrum test block consisting of 12 load levels (Table IX) corresponded to 200 equivalent flight hours (EFH) including ascent, cruise and dive attitudes. The loads were applied to the outside fitting by a numerically programmed hydraulic loading system. Each 200-EFH block required 15 minutes testing time; the entire 24,000 EFH cycle consisted of 120 blocks. Static strain surveys were conducted at 600, 8000, 16,000 and 22,883 EFH intervals. HIP Brace No. 513 withstood the 24,000 EFH spectrum test without failure, thus

LEVEL	LOAD, 10^3 LB		CYCLES/ 200 HR BLOCK
	MAX	MIN	
1	9.0	4.0	1000
2	11.0	-5.0	500
3	14.0	-7.0	1
4	18.0	4.0	50
5	22.0	5.0	9
6	22.0	4.0	15
7	24.0	4.0	7
8	25.0	4.0	13
9	26.0	5.0	2
10	26.0	4.0	2
11	30.0	5.0	2
12	31.0	5.0	1
TEST LENGTH = 24,000 EFH NO. BLOCKS = 120			

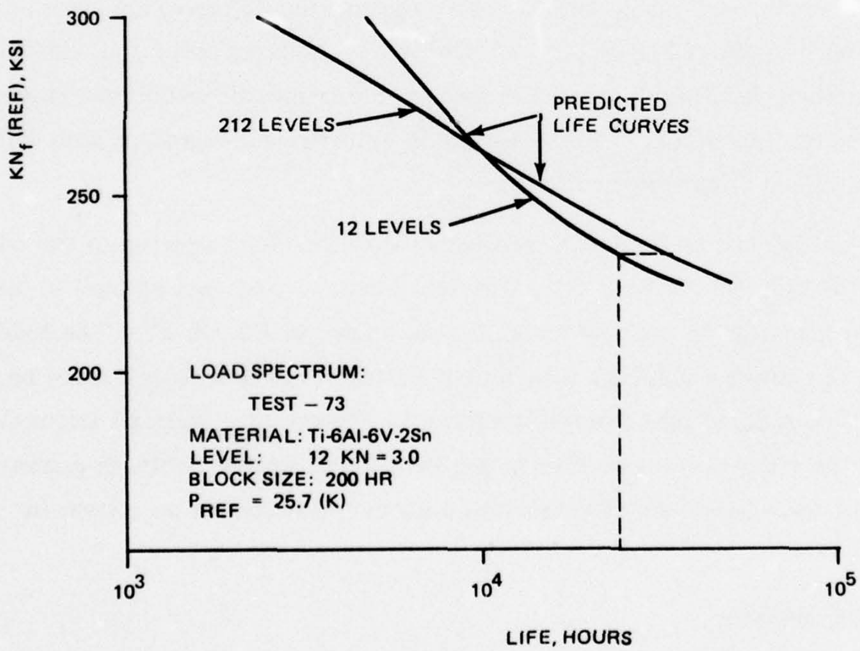


Figure 18. Spectrum Fatigue Life of F-14A Fuselage Brace

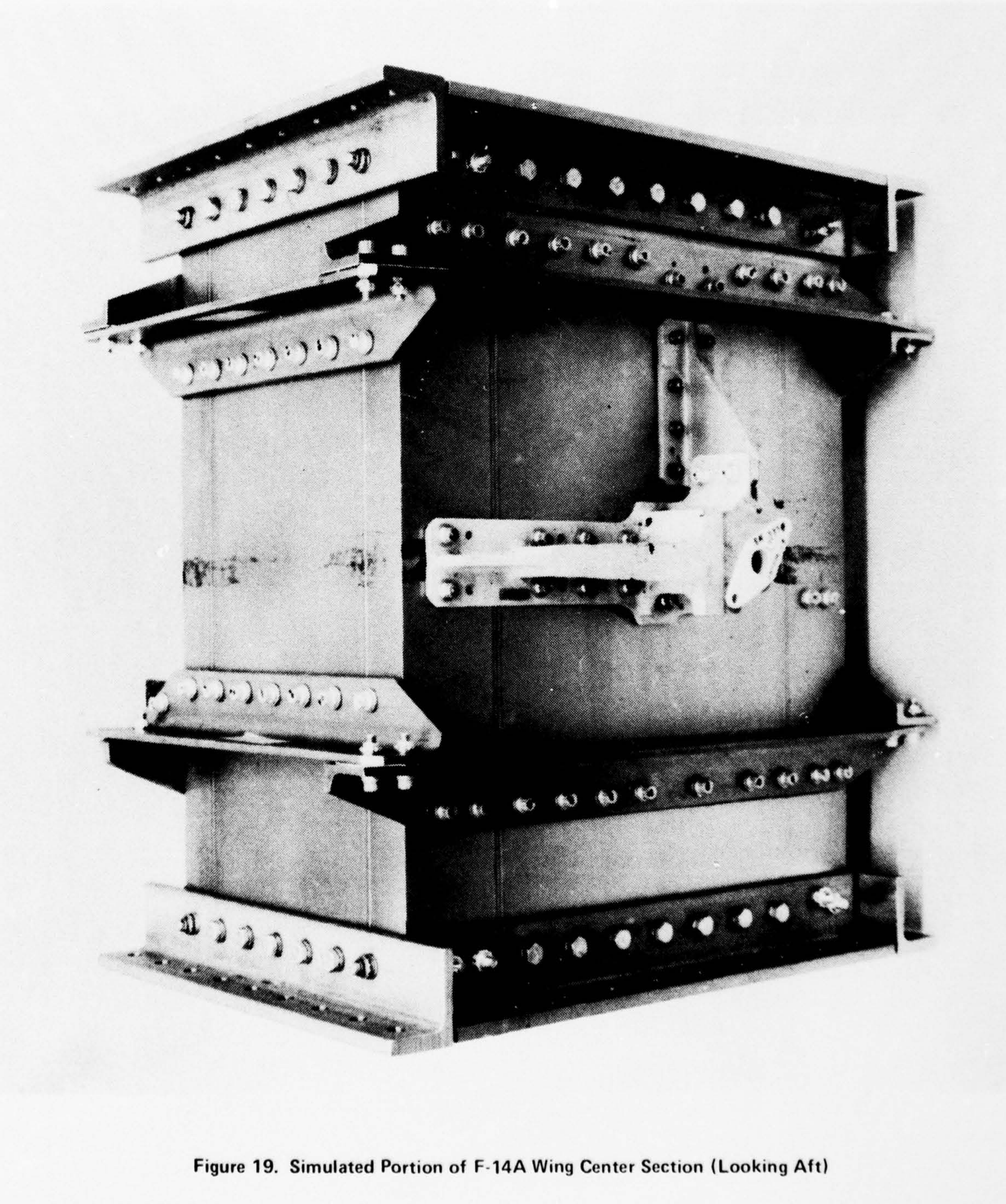


Figure 19. Simulated Portion of F-14A Wing Center Section (Looking Aft)

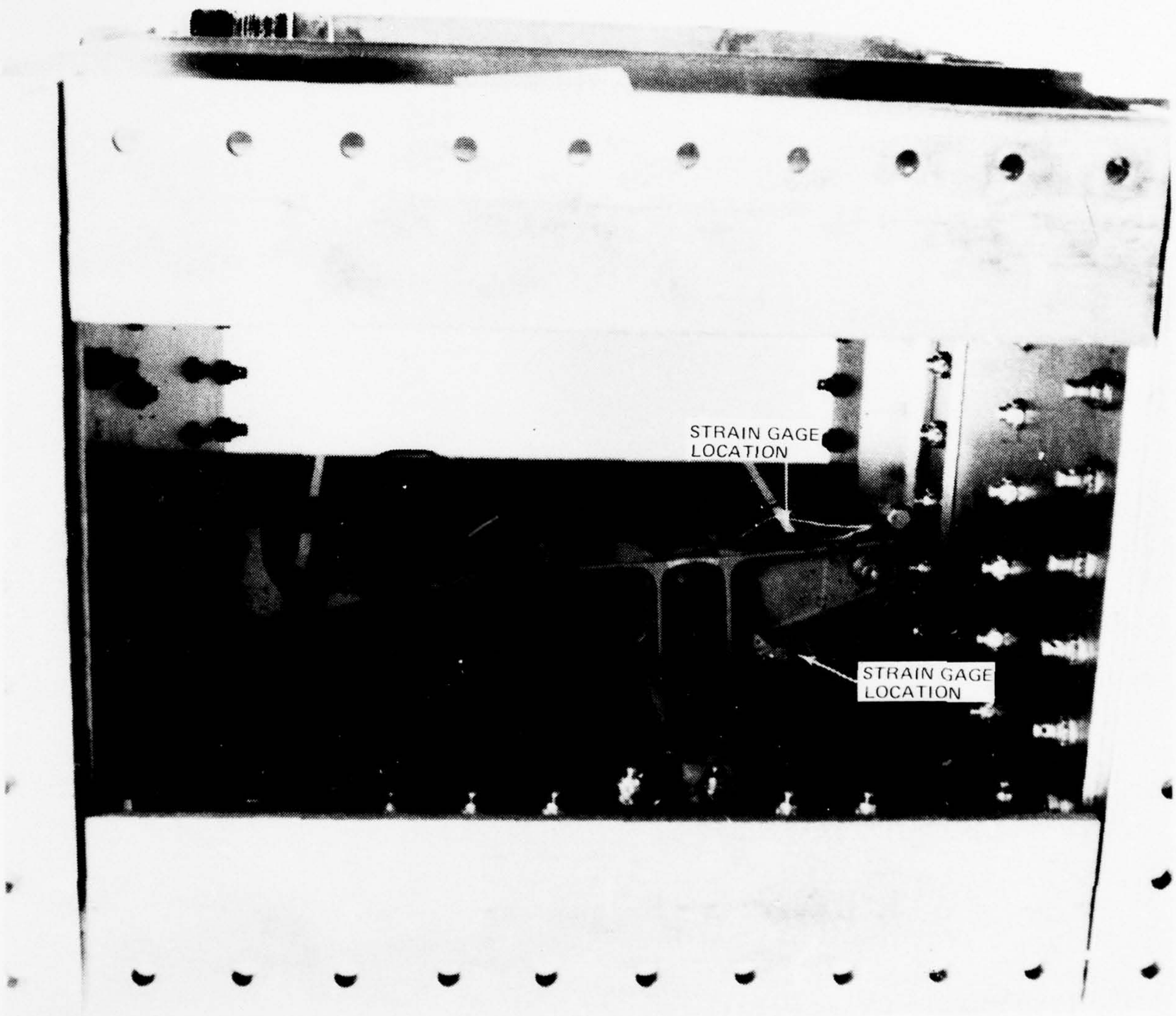


Figure 20. Test Brace Looking Inboard Along Front Beam of Simulated Wing Box

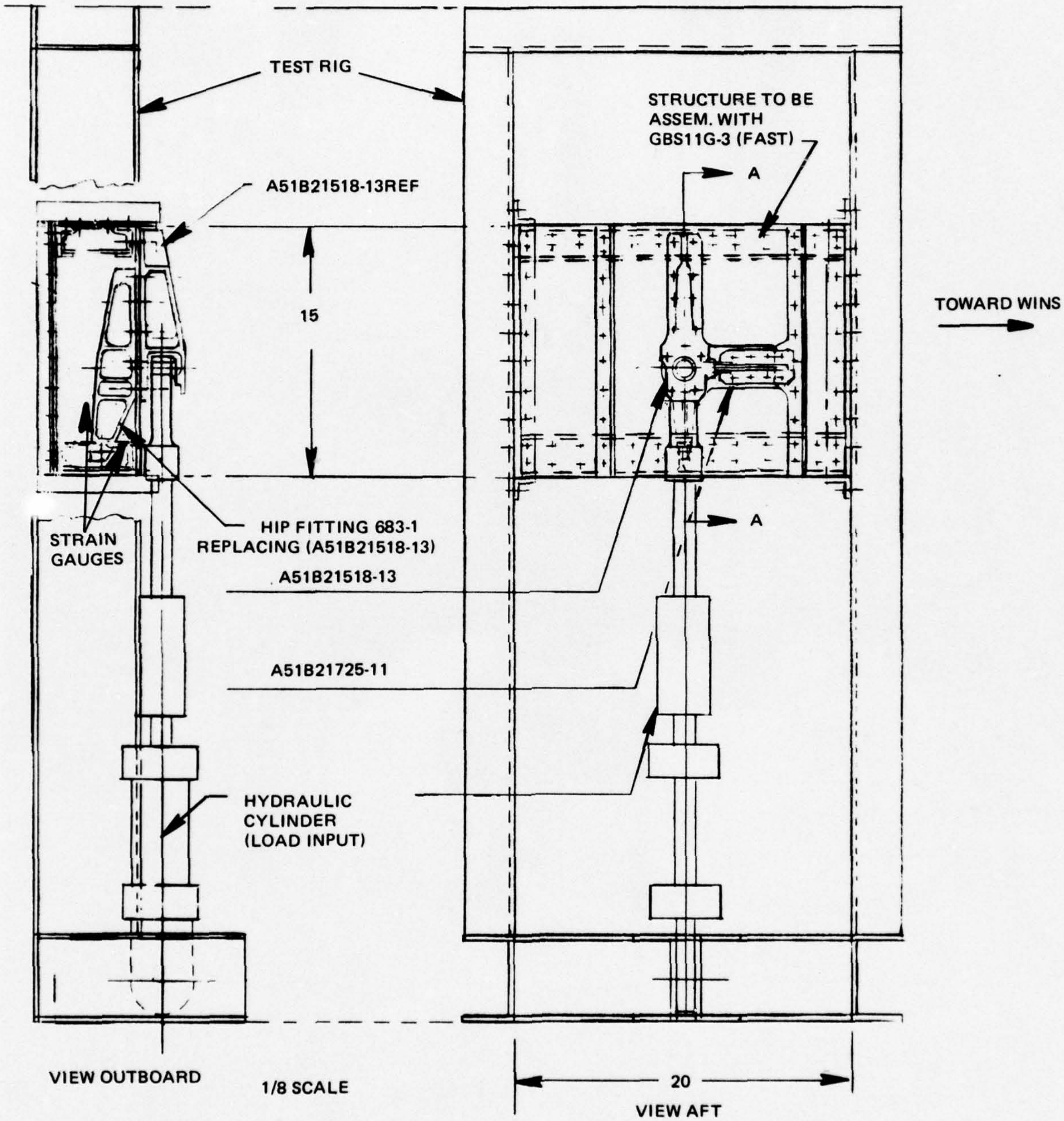


Figure 21. Arrangement for Testing HIP Fitting

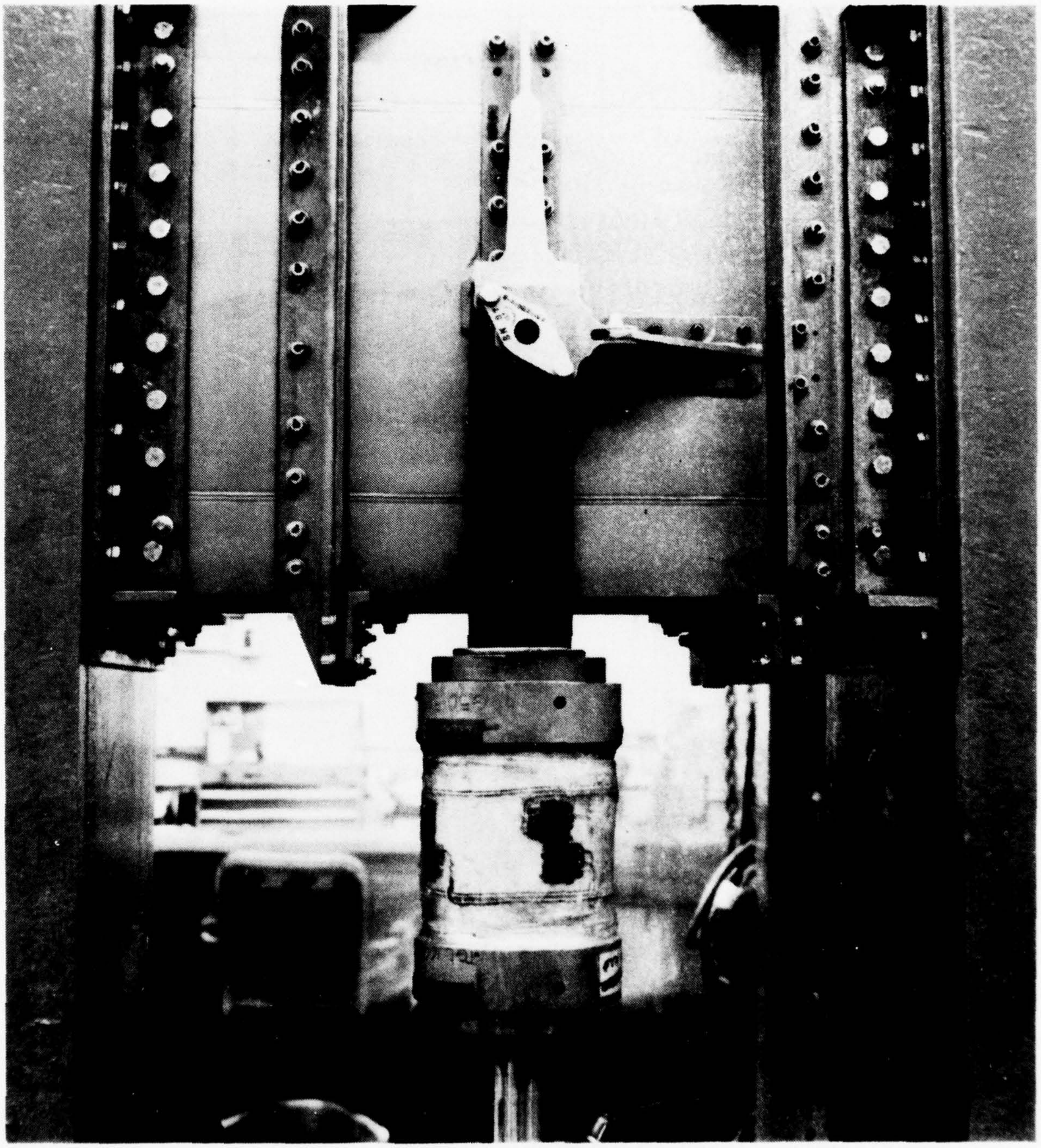


Figure 22. Spectrum Fatigue Testing Fixture

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Table IX SM 513 Spectrum Fatigue Test Results for 24,000 Equivalent Flight Hours with No Failure

LEVEL (2)	LOAD, KIPS		NUMBER OF CYCLES PER 20 OHM BLOCK	FORWARD FLANGE				AFT FLANGE			
				STRAIN, MICROINCHES		STRESS, KSI (1)		STRAIN, MICROINCHES		STRESS, KSI (1)	
	MAX	MIN		FORGED	HIP	FORGED	HIP	FORGED	HIP	FORGED	HIP
1	9	4	1,000	347	387	5.69	6.20	–247	–265	–4.05	–4.24
2	11	5	500	–	–	–	–	–	–	–	–
3	14	7	1	560	610	9.18	9.77	–396	–425	–6.49	–6.81
4	18	4	50	–	–	–	–	–	–	–	–
5	22	5	9	910	951	14.92	15.23	–647	–678	–10.61	–10.86
6	22	4	15	–	–	–	–	–	–	–	–
7	24	4	7	–	–	–	–	–	–	–	–
8	25	4	13	–	–	–	–	–	–	–	–
9	26	5	2	1081	1122	17.73	17.97	–772	–811	–12.66	–12.99
10	26	4	2	–	–	–	–	–	–	–	–
11	30	5	2	–	–	–	–	–	–	–	–
12	31	5	1	1285	1337	21.07	21.41	–915	–970	–15.00	–15.53

1. Based on modulus of 16.4×10^6 for forging, 16.02×10^6 for HIP

2. F-14A Test-73 wing spectrum for Fuselage Brace (Part No. A51B21683).

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establishing the flight-worthiness of fuselage braces (Part No. A51B21683) manufactured by hot isostatic pressing (HIP). Strain survey measurements and corresponding stress values for Brace No. 513 and a control brace produced by conventional methods (forging and machining) are listed in Table IX. Analysis of these data indicated the maximum value of stress encountered in these parts in service to be 21.41 ksi. Accordingly, although the flight-worthiness of selected parts has been established, it was of interest to estimate the true potential of HIP parts by conducting tests at higher stress levels.

3.3 SPECTRUM TESTS AT HIGHER STRESS LEVELS

The test assembly utilized in the qualification tests was modified to allow a more extensive deflection of the front beam, thus inducing higher maximum stresses in the test brace. Strain measurements and corresponding stress levels obtainable by this modification are listed in Table X. The maximum stress level obtained was 32 ksi (forward flange), which was 50 percent higher than the maximum stress experienced by parts in qualification tests. A HIP brace (Part No. 509) was subjected to 120 spectrum test blocks at new stress levels without failure.

3.4 CONSTANT-AMPLITUDE FATIGUE TESTS

Since modifications of the test assembly used in qualification tests resulted only in a limited increase of the maximum stress experienced by the fuselage braces, a new fixture (Figure 23) was designed. The load on the brace in this fixture was applied as shown in Figure 24. Although neither the load application method nor resulting stress distribution corresponded to those in the actual flight article, the new arrangement could allow the tests to continue at the desired stress level to failure. Prior to constant-amplitude fatigue tests, a forged brace was statically loaded in this fixture to determine maximum stresses experienced by the part at various loads; data obtained are listed in Table XI.

The brace used in the static tests was subsequently subjected to constant-amplitude tension-tension ($R=0.1$) fatigue tests at 1800 cpm and 5,000 lb (max) load which corresponds to a maximum stress of 42 ksi in the brace. The part failed after 31,000 cycles by fretting in the loading hole. In subsequent tests, the loading hole was machined oversize and provided with a shrink-fit bushing; the load was decreased to 4000 lb, which corresponds to 34 ksi (maximum) stress in the flange of the brace. The tests conducted on a forged and two HIP braces (Part Nos. 513 and 514) caused failures at 2,700,000, 137,000 and 418,000 cycles, respectively. All failures initiated in the loading hole and exhibited fretting-type characteristics.

Table X Trial Loads in Modified Box

OUTSIDE LOAD, KIPS	FORWARD FLANGE (G 2)		AFT FLANGE (G 1)	
	STRAIN, MICROINCHES	*STRESS, KSI	STRAIN, MICROINCHES	*STRESS, KSI
9	340	5.4	− 230	− 3.7
18	780	12.5	− 570	− 9.1
26	1065	17.1	− 730	−11.7
31	1320	21.1	− 900	−14.7
40	1650	26.0	−1120	−17.9
45	1880	30.1	−1260	−20.2
48	2000	32.0	−1350	−21.6

*Based on modulus of 16.02×10^6 psi

Table XI Trial Loads in Constant-Amplitude Fatigue Fixture

DIRECT LOAD, KIPS	FORWARD FLANGE (G 2)		AFT FLANGE (G 1)	
	STRAIN, MICROINCHES	*STRESS, KSI	STRAIN, MICROINCHES	*STRESS, KSI
1	495	8.1	− 375	− 6.2
2	1005	16.5	− 750	−12.3
3	1500	24.6	−1130	−18.5
4	2060	33.8	−1540	−25.3
5	2540	41.7	−1895	−31.1
6	3100	50.8	−2285	−37.5
7	3610	59.2	−2660	−43.6

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* Forging modulus = 16.4×10^6 PSI

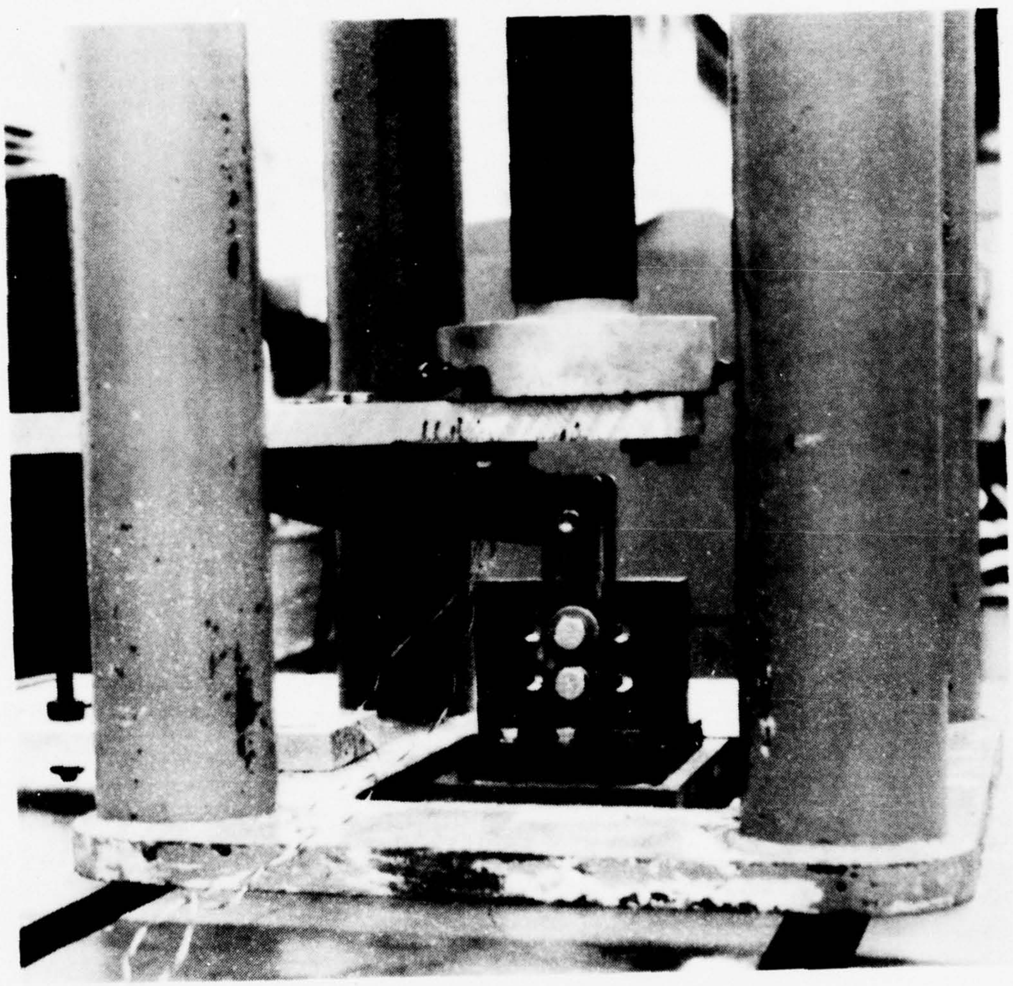

Figure 23. Load-to-Failure Fatigue Testing Fixture

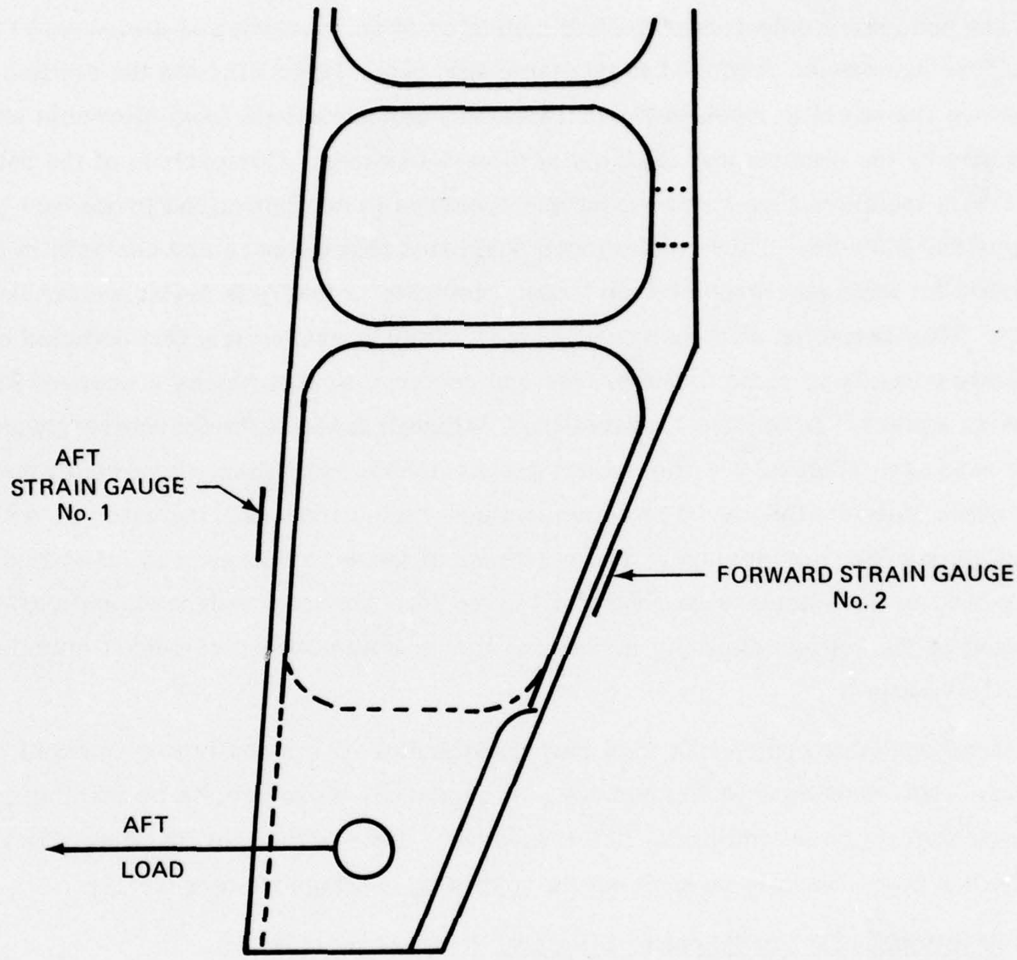

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Figure 24. Section A-A of Figure 3-4

3.5 REPRODUCIBILITY (PILOT LOT) EVALUATIONS

3.5.1 Dimensional Tolerances

The secondary objective of Task 2 consisted of an evaluation of dimensional tolerances of nine fuselage braces produced in the same HIP run. Table XII lists the critical target dimensions (identical to those listed in Table VII) and variations from allowable tolerances determined by the dimensional analysis of pilot-lot braces. Comparison of the data listed in these tables indicated that wider variations occurred in configurations processed in the course of the pilot lot. These differences suggested that temperature controls in the pilot production lot were less precise than those obtainable in the Task 1-Parameter Development studies. This suspicion was confirmed by radiographic evaluations that detected occurrences of isolated porosity in some of the braces and rectangular test blocks processed for material evaluation studies. It became apparent that, although the time/temperature/pressure chart for the autoclave (Figure 25) did not exhibit noticeable anomalies, the actual temperature inside some individually canned mold assemblies varied from that indicated by master thermocouples for the autoclave. The existence of these variations was attributed to an excessive load in the autoclave as shown in Figure 26. The total weight of configurations processed in the reproducibility run was 113 lb. -- far in excess of Task 1 autoclave loadings (8- to 27-lb range).

Metallographic spotchecks indicated that the detected porosity was internal (Figure 27), i.e., not connected to the surface. It appeared, therefore, to be feasible to eliminate these defects by an additional HIP treatment. This method for "healing" the internal porosity has been shown to be effective in upgrading castings (Reference 10).

3.5.2 Additional HIP Treatment to Eliminate Internal Defects

The parts subjected to an additional HIP treatment included five fuselage braces, three test blocks for evaluation of mechanical properties and three machined fracture toughness specimens. The time/temperature/pressure parameters used in this run were 4 hr/1650°F/15 ksi. These shapes were positioned in the autoclave and covered with titanium chips to prevent oxidation; ceramic packings or evacuated enclosures were not utilized. Since there were no thermal insulators around the specimens, no allowances had to be made for soaking time, i.e., the thermal profile for the parts was identical to that of the autoclave.

Subsequent radiography established that the additional HIP treatment completely "healed" porosity in test blocks and machined fracture toughness specimens. Isolated occurrences of surface-connected porosity still remained, however, in fuselage braces. Since location of this porosity was restricted to extremities of the lower base, its effect on the

Table XII Dimensional Reproducibility Studies

DIMENSIONS[1]	TARGET	VARIATIONS FROM ALLOWABLE TOLERANCES[2] [3]									
		SM-506	SM-507	SM-508	SM-509	SM-510	SM-511	SM-513	SM-514	SM-515	
4	4.781	-0.011	WT	WT	WT	WT	-0.005	WT	WT	WT	
5	3.438	WT	WT	WT	WT	WT	WT	WT	WT	WT	
8	0.125	WT	WT	WT	WT	WT	WT	WT	WT	WT	
27	1.515	WT	WT	-0.002	-0.005	-0.005	WT	WT	WT	-0.003	
28	3.140	-0.025	-0.010	-0.025	-0.027	-0.025	-0.010	-0.030	-0.020	-0.025	
30	1.500	WT	WT	WT	WT	WT	WT	-0.010	WT	WT	
31	0.100	(4) WT TO -0.020	(4) WT TO -0.020	(4) WT TO -0.010	(4) WT TO -0.015	(4) -0.006 TO -0.019	(4) -0.003 TO -0.016	(4) WT TO -0.020	(4) WT TO -0.020	(4) WT TO -0.020	
35	2.000	WT	WT	WT	WT	WT	WT	WT	WT	WT	

NOTES:

(1) SEE FIG. A-1 FOR IDENTIFICATION OF DIMENSIONS TAKEN
(2) ALLOWABLE TOLERANCES: +0.030, -0.010 IN.
(3) WT = WITHIN TOLERANCES
(4) RANGE

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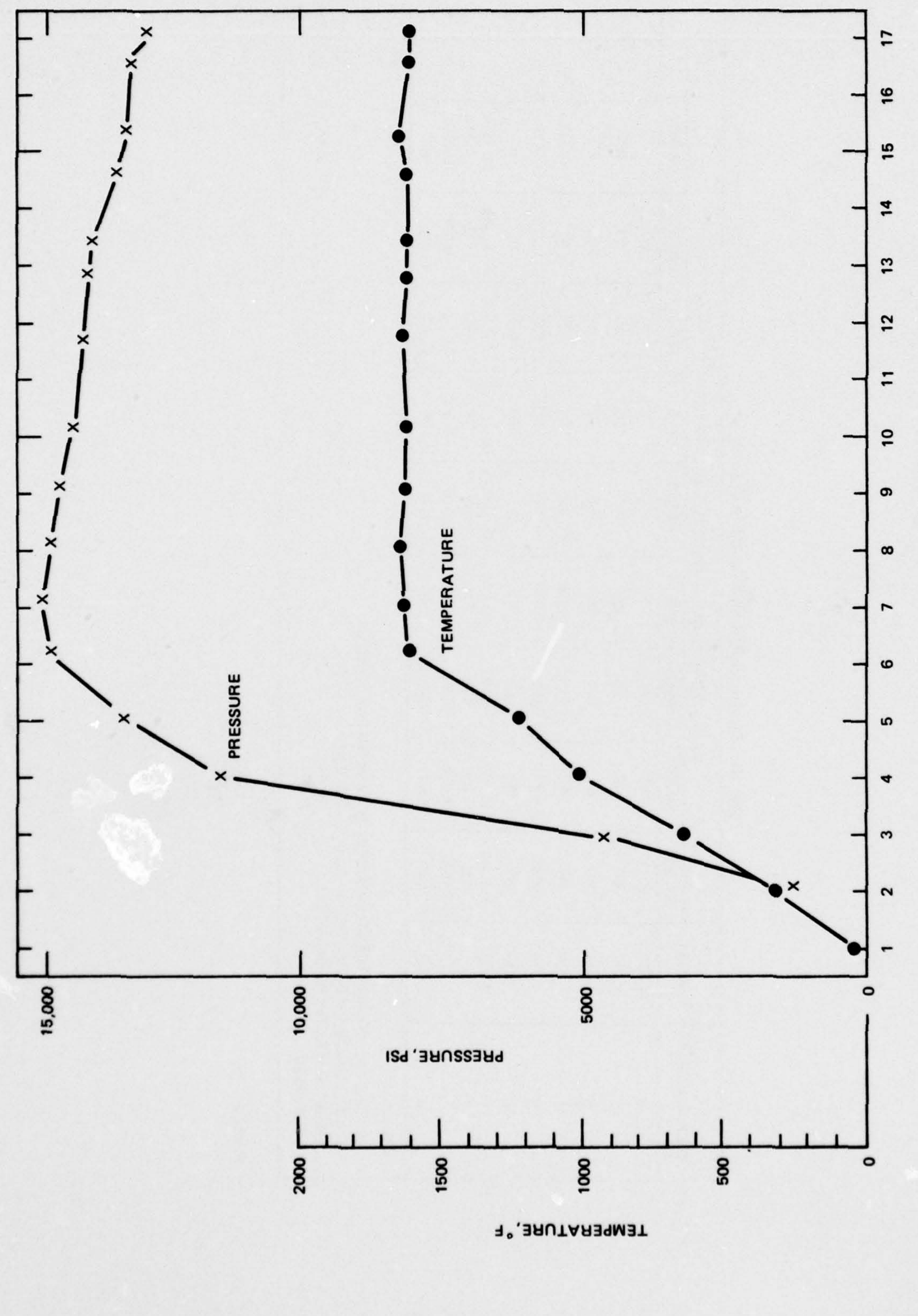


Figure 25. Time-Temperature-Pressure Chart for HIP Pilot Lot (Run 5) Performed at Battelle

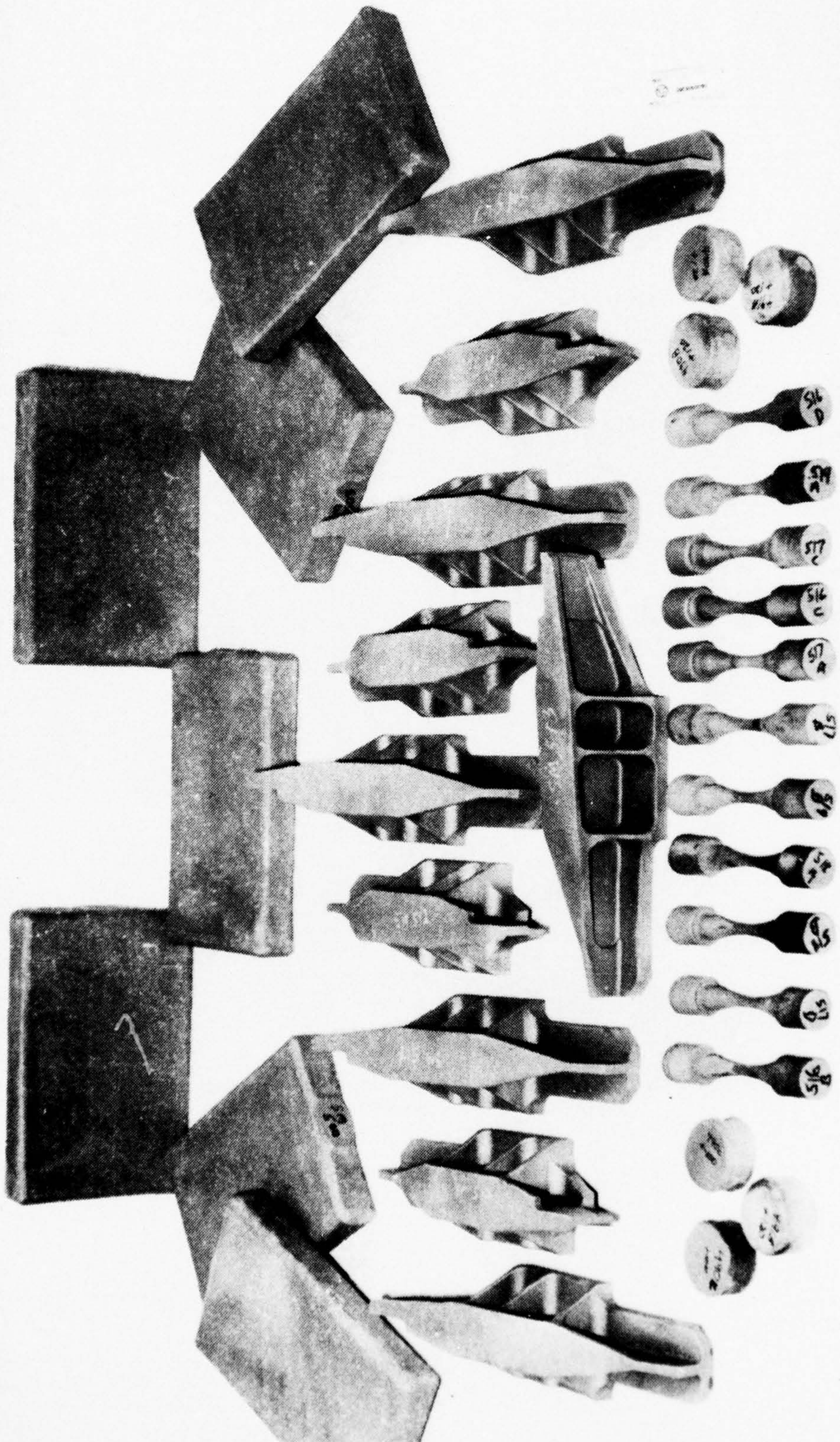
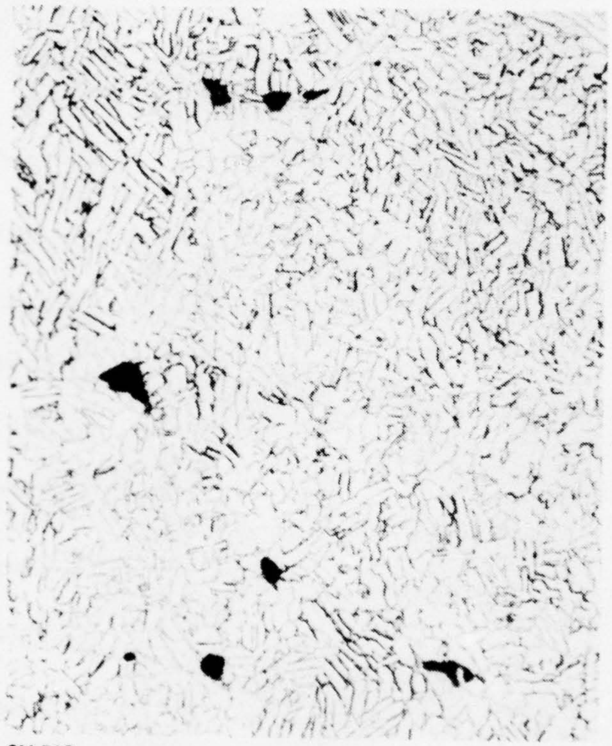


Figure 26. Configurations Processed in the Course of the HIP Pilot Lot (Run 5)



SM 512

Figure 27. Isolated Internal Porosity Detected in Fuselage Brace SM-512 HIP'd in Run No. 5 (290x Mag)

load-bearing capacity was negligible. These defects did not interfere with planned spectrum fatigue tests.

3.5.3 Refinement of Temperature Controls in HIP Autoclave

Apparent temperature gradients detected in the course of reproducibility studies indicated that the temperature of each individual mold assembly must be controlled to ascertain that the required temperature is reached and maintained during the HIP cycle. One approach to attain this objective is to weld a thermocouple to the metallic enclosure of the mold assembly. The feasibility of this approach was established in HIP Run No. 7, which produced a sound fuselage brace (SM 558) without external or internal defects in a fully loaded autoclave.

Temperature data automatically recorded for the thermocouple welded to the outer (steel) envelope of the mold assembly indicated the temperature to be within $-0/+8^{\circ}F$ of the required setting. Dimensional checks indicated the thickness of the center web (Dimension No. 31, Table XII), which is one of the principal load-carrying members of the structure, to be now well within required tolerances.

3.5.4 Verification of Mechanical Properties

3.5.4.1 Tensile Properties

Representative test data on specimens machined from rectangular test blocks and braces are listed in Table XIII. As discussed in previous sections, five braces from the pilot lot were subjected to an additional HIP and vacuum annealing cycle. Accordingly, two sets of data are presented: one for "As-HIP" and one for "As-Re-HIP" materials. Although yield strength values for Re-HIP braces were approximately 4 ksi lower than corresponding values for braces subjected to a single HIP treatment, this variation was judged statistically insignificant. All ultimate and yield strength values were within 5 percent of the current requirements for annealed forgings. Typical elongation properties exceed applicable specification minimum requirements by a factor of two.

It is apparent that ultimate and yield strengths of HIP materials can be increased to meet current requirements for forgings with minor modifications in the oxygen content within the allowable limits, without detrimental effects on their excellent elongation characteristics and fracture toughness. Replacement of tungsten electrodes with (consumable) titanium electrodes in powder manufacturing processes and upgrading quality control procedures to ensure freedom from foreign inclusions will also contribute to an improvement in mechanical characteristics.

3.5.4.2 Fatigue Endurance

Fatigue endurance data for specimens manufactured in HIP Run No. 5 and subjected to an additional HIP cycle (followed by vacuum annealing at 1300°F) are listed in Table XIV. When comparing these data values with Task 1 fatigue endurance data presented in Figure 5, it is evident that repeated HIP treatment resulted in a wider scatter of experimental data. Accordingly, at the present time repeated HIP cycling cannot be recommended for processing of Ti-6Al-6V-2Sn titanium alloy parts for fatigue critical applications. Limited data on saturation shot peening to 0.1 Almen intensity indicate that fatigue properties can be significantly improved by this treatment as shown in Table XIV.

3.5.4.3 Fracture Toughness

Three fracture toughness specimens, which were machined from re-HIP'd and vacuum annealed blocks, yielded K_{1c} values of 72, 73 and 74 ksi $\sqrt{\text{in,}}$ thus confirming the superior fracture toughness characteristics of HIP materials. The obtained values were comparable to findings of Phase 1 evaluations. Extraordinarily high fracture toughness characteristics of HIP materials are believed to be related to isotropic characteristics of the microstructure which cause frequent changes in the direction of the fracture-path and thus retard the crack propagation, as discussed in Section 2.4.5.

3.6 ELECTRON-BEAM (EB) WELDABILITY EVALUATIONS

Electron-Beam (EB) welding has a far-reaching and promising potential in the design of large hybrid components from net or near-net configurations manufactured by HIP. EB welding is especially well suited for manufacturing of hybrid components, since the depth of weld penetration obtainable by commercial processes exceeds 2 in. for titanium alloys, preparation of faying surfaces is relatively simple and inexpensive, and utilization of vacuum chambers virtually excludes the possibility of contamination.

Initial welding experiments with HIP'd Ti-6Al-6V-2Sn titanium alloy blocks resulted in extensive porosity. Subsequent experiments on identical material after vacuum annealing at 1300°F resulted in high-quality welds as shown in Figure 28 for 2-in.-thick material. These welds met all applicable ultrasonic and radiographic acceptance criteria. The difference in the obtainable quality of welds is attributable to the variation in the hydrogen content of HIP parts. Even in properly outgassed ceramic molds, the expected hydrogen pickup on HIP processing is in the range of 300 ppm and leads to extensive porosity in EB welds. Vacuum annealing at 1300°F results in lowering of the hydrogen content to below 100 ppm, at which level porosity problems are no longer encountered.

Table XIII Tensile Properties of Configuration Processed in the Course of Reproducibility Studies

SPEC ID	PARENT CONFIGURATION	CONDITION	F_{tu}, KSi	F_{ty}, KSi	ELONG %
69	TEST BLOCK	AS HIP + Vac Ann.	145.5	137.4	17.0
70	TEST BLOCK	AS HIP + Vac Ann.	145.5	137.6	16.5
71	TEST BLOCK	AS HIP + Vac Ann.	150.2	141.2	16.0
72	TEST BLOCK	AS HIP + Vac Ann.	150.2	140.2	17.0
513-1	BRACE 513	AS HIP + Vac Ann.	147.4	137.6	18.0
513-2	BRACE 513	AS HIP + Vac Ann.	149.2	139.1	18.0
101	TEST BLOCK	AS RE-HIP + Vac Ann.	145.5	141.9	15.0
102	TEST BLOCK	AS RE-HIP + Vac Ann.	144.9	141.8	15.5
103	TEST BLOCK	AS RE-HIP + Vac Ann.	146.7	139.4	16.0
104	TEST BLOCK	AS RE-HIP + Vac Ann.	148.1	141.4	15.0
509-1	BRACE 509	AS RE-HIP + Vac Ann.	145.3	135.5	18.0
509-2	BRACE 509	AS RE-HIP + Vac Ann.	142.0	133.2	18.0

5287-35

Table XIV Fatigue Endurance of Re-HIP'd Ti-6Al-6V-2Sn Configurations

SPECIMEN NUMBER	MAXIMUM CYCLIC STRESS		CYCLES TO FAILURE	REMARKS
	KSI	MPa		
P1	100	689	48,000	
P2	90	621	78,000	
P3	80	552	565,000	
P4	70	483	451,000	
P5	65	448	905,000	
P6	60	414	896,000	
1	65		3,206,000	SATURATION SHOT PEENED TO 0.1 ALMEN
2	75		1,835,000	SATURATION SHOT PEENED TO 0.1 ALMEN

5287-36

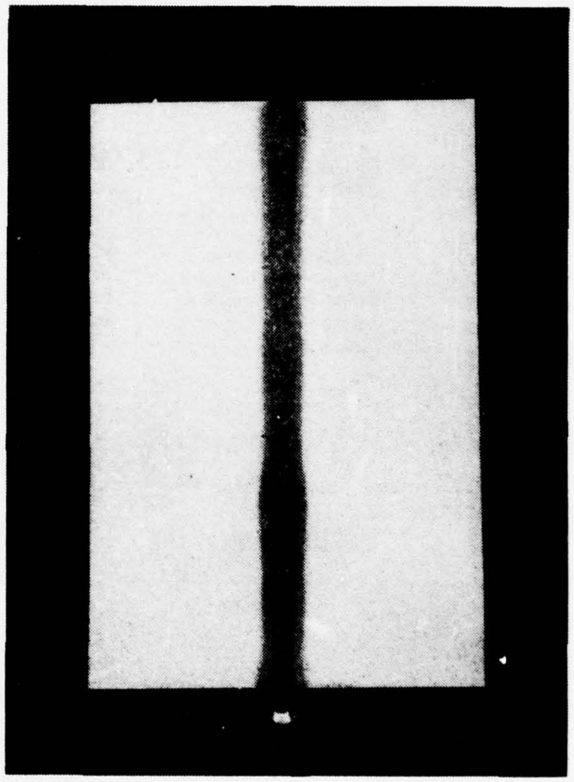


Figure 28. EB Butt Weld in 2-Inch-Thick HIP'd Ti-6Al-6V-2Sn Section (1.6x Mag)

Excellent mechanical properties were indicated by tests conducted on experimental transverse butt welds. Tensile ultimate strength, yield strength and elongation for the EB butt weld joint were 148.5 ksi, 138.3 ksi and 16.0%, respectively. Additional studies to complete the weldability evaluations should have included investigations of fracture toughness, fatigue and stress-corrosion characteristics. These studies were, however, beyond the scope of this program.

Section 4

ACCEPTANCE CRITERIA

4.1 INTRODUCTION

Typical defects encountered in HIP materials differ significantly from those encountered in wrought and cast materials, and their detection requires modification and/or refinement of existing NDI methods or development of entirely new techniques.

The two types of defects of major concern in establishing the acceptance criteria for HIP materials are: (1) Fine porosity remaining on incomplete densification, and (2) Presence of foreign inclusions introduced either during powder manufacturing or subsequent processing. Detectability of both types was studied by various methods, including the radiographic image-enhancement techniques and measurement of ultrasonic velocity in porosity-containing materials.

4.2 IMAGE PROCESSING SYSTEM

4.2.1 Background

The Image Processing System used in this study is a combination of hardware and software interfaces which have been in development since 1970. Figure 29 represents the imaging system in a block-flow format. All images processed by the system are viewed through the Vidicon camera. These images are either analyzed by the digital section (left-hand side of the diagram) or the hardware section (right-hand side of the diagram) of the image-processing system. The present study utilized the edge refinement, color assignment and magnification capabilities of this system.

4.2.2 System Capabilities

4.2.2.1 Edge Refinement

In edge refinement, the edges of images are emphasized and backgrounds eliminated for easier recognition of boundaries, lines and fine structures. Edge refinement is performed by the analog computer portion of the Image Processing System. The computer permits the derivative of the video signal to be measured and visually displayed on the television monitors. This derivative signal can be mixed with the normal video signal in varying degrees for optimum analysis. Edge-line widths can be controlled and adjusted from thick to narrow for maximum visibility of fine details. Images may be refined during positive or negative viewing.

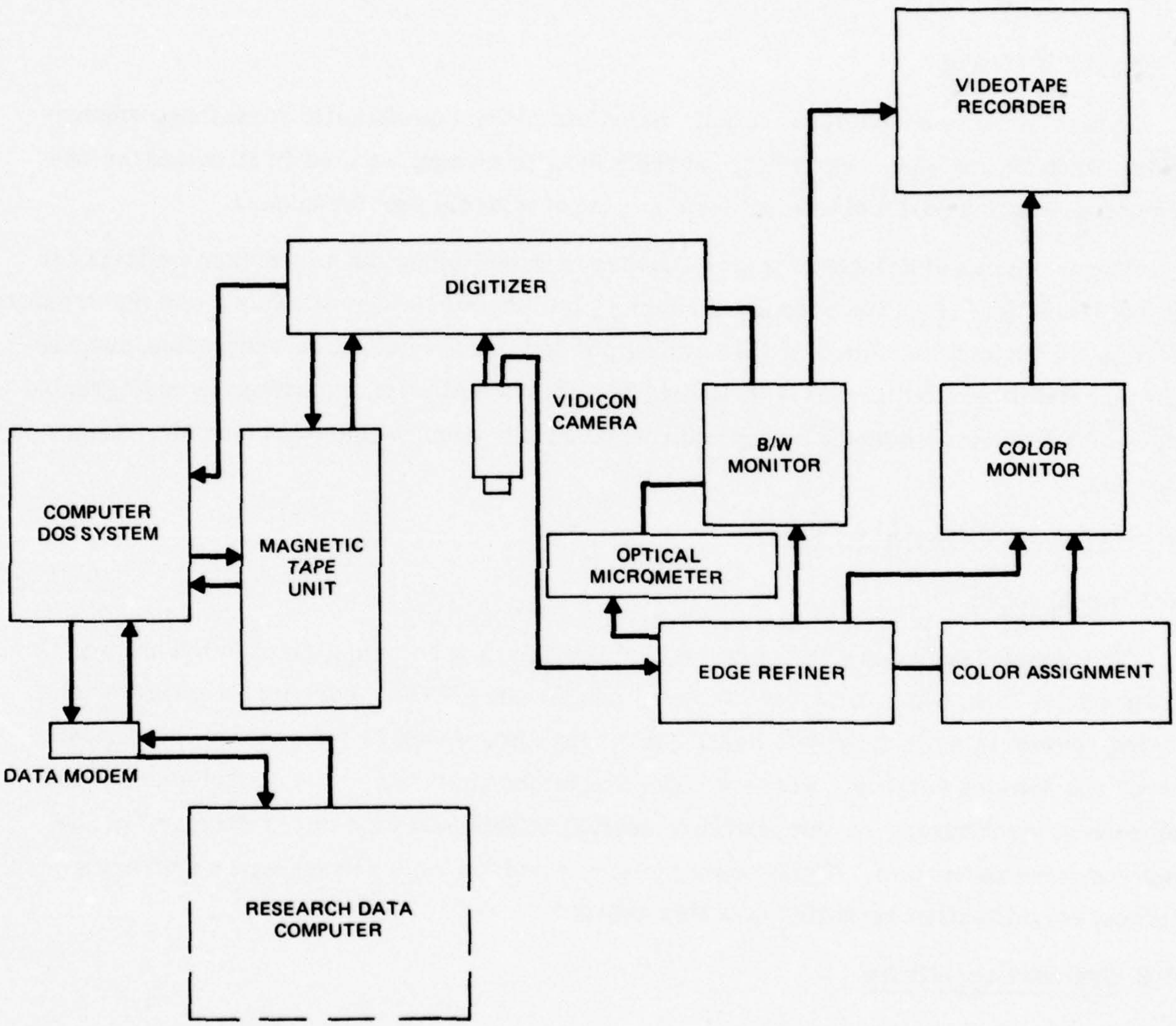


Figure 29. Diagram of the Total Concept Image Processor

4.2.2.2 Color Assignment

When color assignment is applied, colors are assigned to density ranges to permit easy identification of contours and density boundaries. Color assignment is performed by a logarithmic circuit that electronically analyzes the photographic density of the image being viewed and classifies the densities into 12 discrete colors. These colors allow density contours to be analyzed and, when performed in combination with edge refinement, border areas are further accentuated.

4.2.2.3 Magnification

The magnification capabilities of the Image Processing System are controlled by the front-end optics. The system can view an image from radiographic to metallographic samples using reflected light sources. The extent to which a radiograph can be magnified is dependent on its grain size and a factor termed "magnification resolution".

4.3 UTILIZATION OF THE IMAGE-ENHANCEMENT SYSTEM

4.3.1 Detection of Isolated Porosity

For materials up to 0.300-in. thick, detection of porosity of 0.005 in. and larger in diameter by standard radiographic techniques presented no problems. By using finer grain films and increasing the exposure time while decreasing the intensity of radiation, the limit of detectability could be widened to include 0.003-in.-diameter pores. By inspecting suspected areas of the film by the image enhancement system utilizing edge refinement and magnification techniques, porosity with diameters as low as 0.001 in. could be detected.

4.3.2 Tungsten Particle Inclusion

Titanium alloy powders manufactured by the REP process using tungsten electrodes frequently contain tungsten inclusions. Although the number and size of these inclusions could be significantly decreased by modifications of the electrode configurations, the REP powder grades available at the time this program was initiated still contained isolated tungsten inclusions. Contrast between radiographic images of high-density tungsten particles and the matrix permitted a reliable detection of tungsten particles down to 0.003 in. in size using fine-grained films and optimized exposure parameters. Detectability of fine tungsten particles could be further improved by the image-enhancement system. Only limited data were generated, however, since recent modifications of the process by Nuclear Metals included a replacement of tungsten electrodes by consumable titanium electrodes; thus, eliminating the source of the problem.

4.3.3 Other Foreign Inclusions

Metallographic evaluations of consolidated materials also detected the presence of silicon- and aluminum-containing particles. Although the presence of oxygen cannot be confirmed by standard microprobe techniques, the particles are believed to be oxides of these elements. These contaminants were apparently introduced during the powder manufacturing process, since they were detected in configurations consolidated in both ceramic and metal molds. The contrast between radiographic images of these particles and the metal matrix was much less pronounced than that produced by tungsten inclusions. Accordingly, detection of ceramic particles by radiographic techniques is not fool-proof, even when optimized radiographic parameters are utilized, including the image-enhancement system. Metallographic evaluations must be relied on to ascertain the absence of these inclusions.

Recent reports indicate that Nuclear Metals has established the quality control procedures required to ensure freedom from ceramic inclusions. Once confirmed by metallographic and statistically valid evaluations, these provisions will definitely facilitate development of practical and cost-effective acceptance standards.

4.4 MEASUREMENT OF ULTRASONIC VELOCITY

Uniformly distributed (bulk) microporosity experienced in incompletely densified powder metallurgical configurations may not be detectable by conventional NDI techniques. Initial feasibility studies indicated an inverse relationship between remaining microporosity and velocity of 5-MHz ultrasound in configurations consolidated from hydride/dehydride type Ti-6Al-4V titanium alloy powder. Verification of this relationship for configurations consolidated by HIP from REP Ti-6Al-6V-2Sn titanium alloy powders was one of the objectives of Task 3 of this program. The specimens for this evaluation were consolidated by HIP from -35, +200, +120 and +100 mesh sizes.

The volume percent of remaining porosity was estimated based on metallographic evaluation of representative cross-sections and determination of area fractions occupied by porosity images. Data on variations of ultrasound velocity versus density of 3/4-in.-thick test blocks are presented in Figure 30. These findings verified the direct relationship between the ultrasonic velocity and density of HIP Ti-6Al-6V-2Sn titanium alloy configurations consolidated from REP powders. Generated data were obviously insufficient to provide for a statistical evaluation of this relationship. They did indicate, however, that this method may prove to be a powerful tool in establishing quality control criteria for HIP processing.

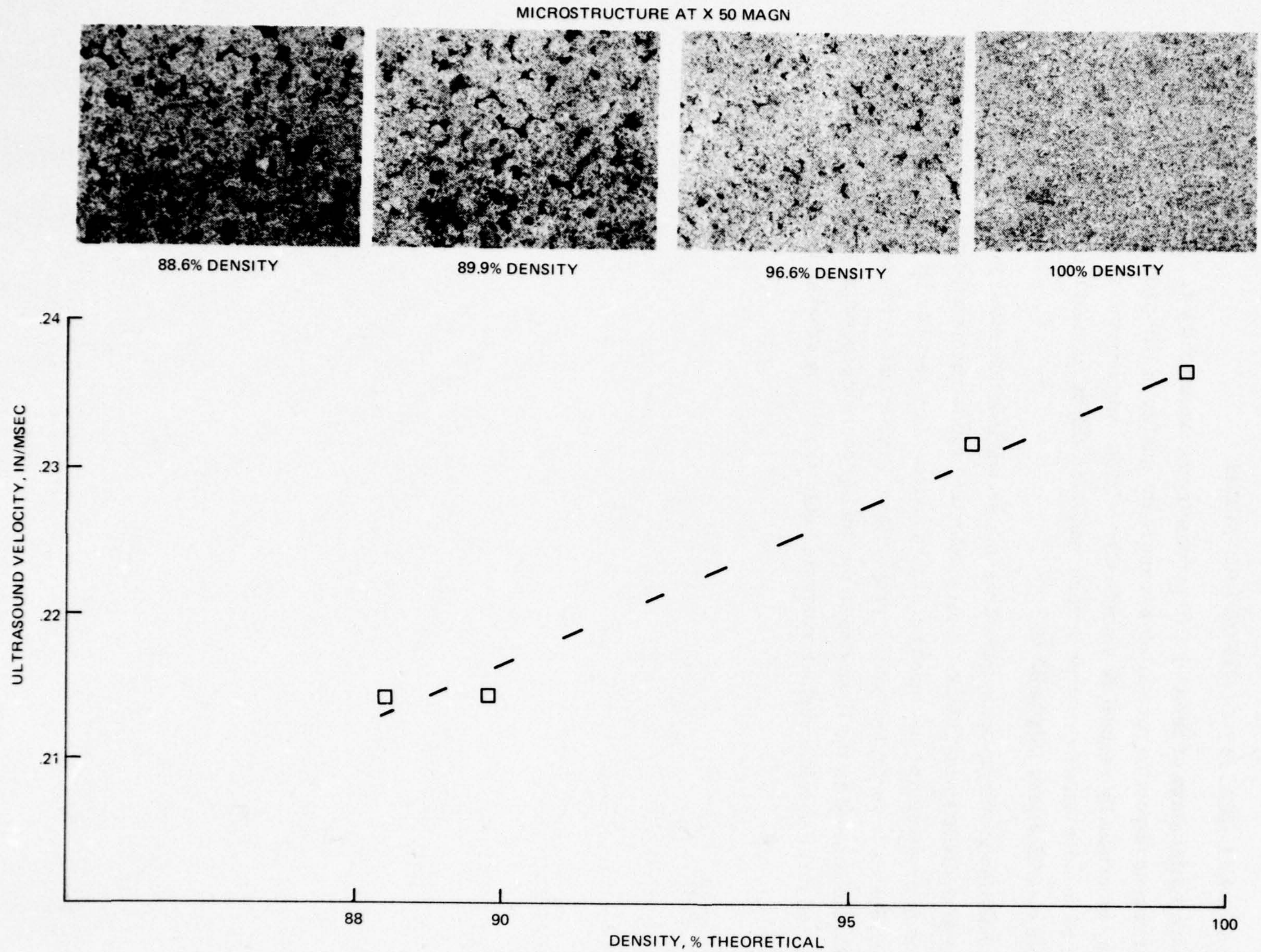


Figure 30. Relationship Between 5-MH_z Ultrasound Velocity and Density of HIP'd Ti-6Al-6V-2Sn Configuration

5287-1

4.5 ADDITIONAL QUALITY CONTROL PROVISIONS

In the initial stage of applying HIP processing in manufacturing, it will be essential to utilize stringent inspection provisions to assure the quality of HIP products. These provisions may be gradually relaxed as statistically reliable data become available on the effectiveness of vendors' quality control criteria and their ability to meet the requirements of applicable specifications (Appendix E).

In addition to radiographic and ultrasonic evaluations described in previous sections, processing of auxiliary test blocks in each autoclave run is recommended to perform spot-checks on microstructure and mechanical properties. One part per lot should also be sectioned and tested to verify mechanical properties. Cost-effective fluorescent penetrant techniques should be used as the first step in the quality control procedure to detect surface porosity and other possible surface-connected defects such as cracks, laps and pits.

Section 5

SUMMARY

The primary objective of this program was to verify the flight-worthiness of F-14 fuselage braces (Part No. A51B21683) manufactured by hot isostatic pressing (HIP) by performing spectrum fatigue tests simulating four design life cycles. These tests were conducted in a test assembly simulating the actual section of the F-14 wing-box structure in which fuselage braces are installed during assembly operations. One test brace was tested and withstood spectrum tests without failure. All spectrum stress levels were subsequently increased by 50 percent. One additional fuselage brace was tested and withstood increased stress levels without failure.

Other objectives of this program were: (1) verification of mechanical properties of HIP materials, (2) evaluation of dimensional reproducibility of the pilot lot, and (3) identification of applicable NDI criteria.

HIP materials exhibited excellent fracture toughness and elongation characteristics, exceeding by far the design specifications. Some values for tensile and yield strength were approximately 3 percent below the current design specifications for annealed forgings. It is believed, however, that the required values can be met by a comfortable margin by minor modifications of the oxygen content in the original powder (1800 ppm) within allowable specifications (2000 ppm maximum). Furthermore, even at the present strength level, isotropy of material virtually assures its applicability in most Ti-6Al-6V-2Sn titanium alloy airframe structures.

Although current tight specifications for machined components could be met by critical members of the fuselage brace configuration, minor deviations from specified values were encountered for some non-critical members. At the present time, there appear to be two alternative approaches to resolve this problem: (1) to consider relaxation of currently tight tolerances for some non-critical members or (2) to perform additional refinements of the mold configuration to ascertain that all dimensions will fall within currently required tolerances. In view of the relatively low stresses detected in spectrum fatigue tests, the minor degree of variations encountered and cost-effectiveness considerations, the first approach is preferable.

Studies of NDI acceptance criteria included evaluations of radiographic image-enhancement techniques for detection of isolated porosity and inclusions, and ultrasonic velocity

measurements to detect fine (bulk) porosity in incompletely densified materials. Utilizing the edge-refinement and magnification capabilities of the image-enhancement system, resolution of 0.001 in. porosity and 0.003 in. tungsten inclusions was shown to be feasible. An inverse straight-line relationship was indicated between the percent of remaining microporosity and the velocity of 5-MHz ultrasound.

Very precise control of HIP parameters is required to assure quality of the product. Certification of powder grades to be free from foreign inclusions and enforcement of rigid specifications pertaining to mold-manufacturing processes, allowable autoclave loads and temperature controls for individual mold assemblies are of paramount importance.

Tensile yield and elongation characteristics of electron-beam welds were comparable to those of the parent metal. These welds also met all radiographic and ultrasonic acceptance criteria.

A set of specifications has been proposed for powder manufacturing and HIP operations and requirements for the final product.

Section 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

- Fuselage braces (Part No. A51B21683) manufactured by the Hot Isostatic Pressing (HIP) process withstood F-14A Wing Box spectrum tests simulating 24,000 equivalent flight hours (four design lives) without failure.
- Projected savings related to the utilization of the HIP process in manufacturing of these parts are in the range of 30 to 40 percent.
- Microstructure of HIP configurations exhibited completely isotropic characteristics.
- Vacuum annealing at $1300^{\circ}F$ is essential to ascertain acceptable mechanical properties and electron-beam weldability of HIP-processed Ti-6Al-6V-2Sn titanium alloy.
- Fracture toughness K_{1c} values were in the range of 69 to 74 ksi $\sqrt{in.}$ and significantly exceeded corresponding values for annealed forgings, typically 57 ksi $\sqrt{in.}$ Improvement of fracture toughness characteristics is believed to be associated with the diversion of the crack tip propagation at former grain boundaries.
- Elongation was in the range of 15 to 16 percent and thus exceeded the specified value for forgings (8 percent) by a factor of two.
- At the present oxygen level (1800 ppm), the values for F_{tu} and F_{ty} were approximately 3 percent below the design specifications. Specified values for F_{tu} and F_{ty} (150 ksi and 140 ksi, respectively) can be obtained, however, by minor adjustment of the oxygen content within the allowable limits.
- Fatigue endurance limit of HIP'd Ti-6Al-6V-2Sn titanium alloy in tension-tension (R=0.1) is in the range of 70 to 80 ksi and thus comparable to that of annealed plate.
- Precise control of parameters is of utmost importance in processing of Ti-6Al-6V-2Sn titanium alloy powders. Freedom from temperature gradients and proper outgassing of ceramic molds and packing materials warrant particular attention.
- Nondestructive inspection (NDI) capabilities can be greatly improved by utilization of radiographic image-enhancement techniques.

- Detection of isolated fine foreign ceramic particles can be ascertained at the present time only by extensive metallographic studies. Availability of certified inclusion-free powders is an essential prerequisite for utilizing the HIP process in manufacturing of highly stressed airframe components.
- Existing tight tolerances for machined components may have to be relaxed to ascertain that all dimensions of HIP parts fall within design specifications, thereby ensuring that the significant cost saving potential is being fully realized.

6.2 RECOMMENDATIONS

- Studies should be initiated to evaluate the feasibility of utilizing the HIP process in combination with EB welding in manufacturing of hybrid critical airframe components such as nacelle frames and bulkheads. The potential savings projected for these parts are in the order of \$1,000,000 per lot of 50 aircraft.
- Although currently available REP powder grades are suitable for initial feasibility studies, verification of mechanical and physical properties will require use of certified inclusion-free powders. The processes for manufacturing such powders are currently in final stages of development by several vendors, including Nuclear Metals, Inc.

Section 7

REFERENCES

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10. J. Magnuson, "Repair of Titanium Airframe Castings by Hot Isostatic Pressing," Grumman Report ADN 11-06-75.6

Appendix A

GRUMMAN F-14A FUSELAGE BRACE ENGINEERING DRAWING

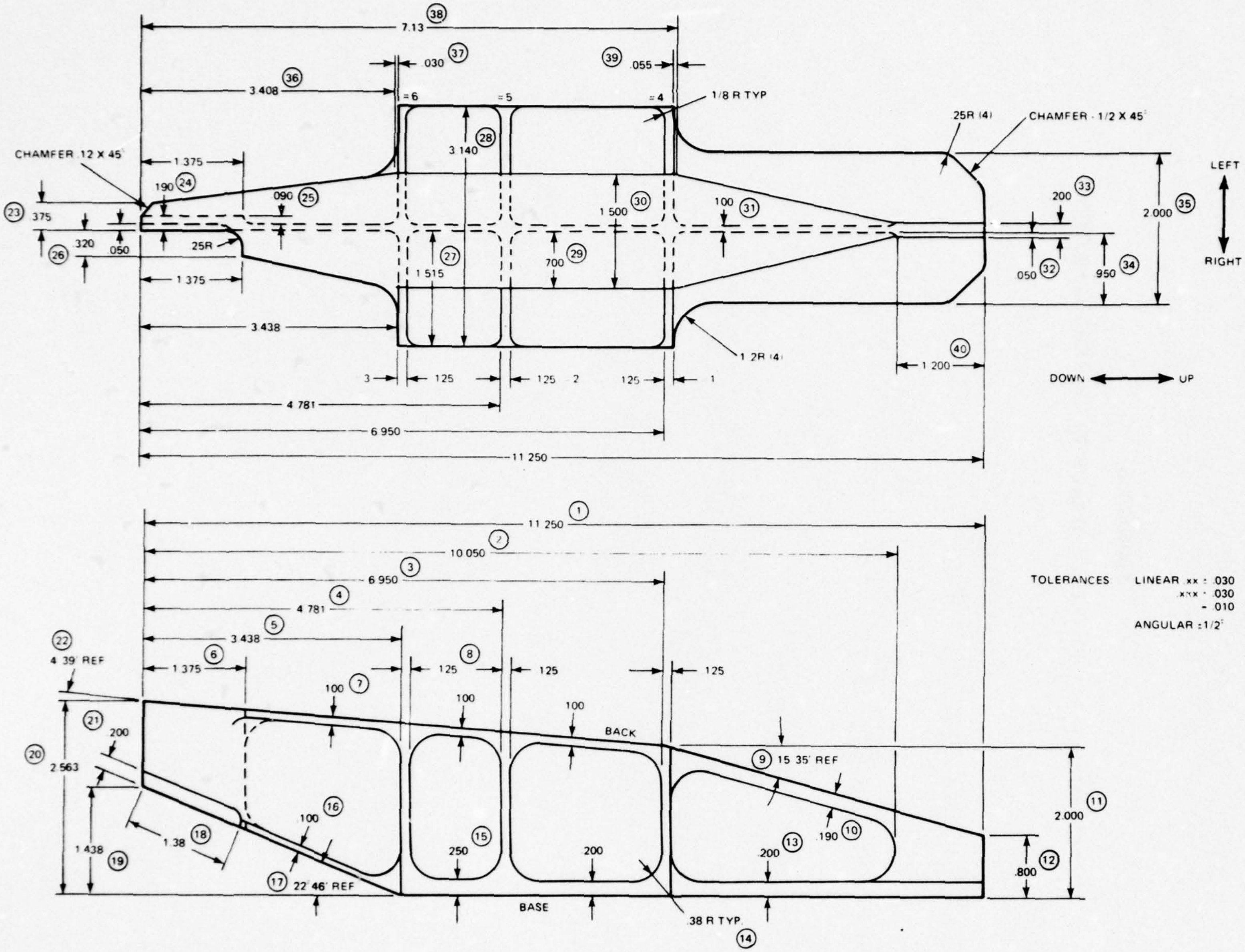


Figure 31. Grumman Engineering Drawing for Part No. A51B21683 (Fuselage Brace)

Appendix B

TEST COUPON CONFIGURATIONS

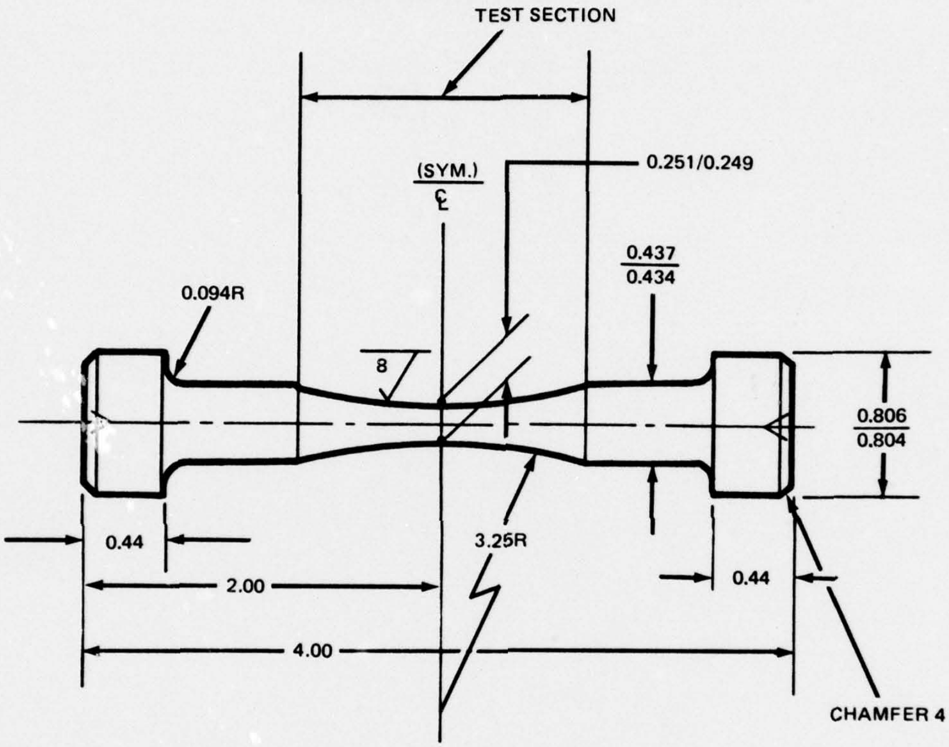
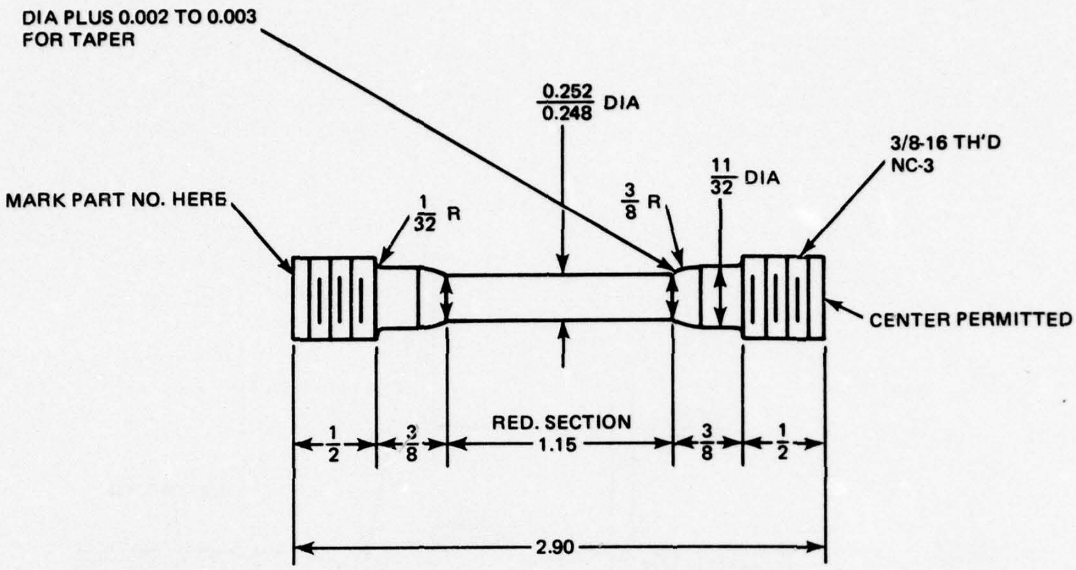
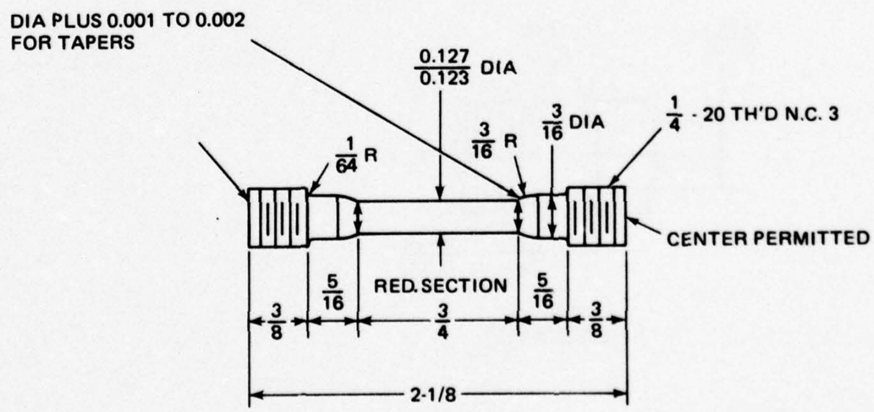


Figure 32. TGS 5771 Fatigue Specimen



A. TGS 5307



B. TGS 5303

Figure 33. Tensile Specimen Configurations

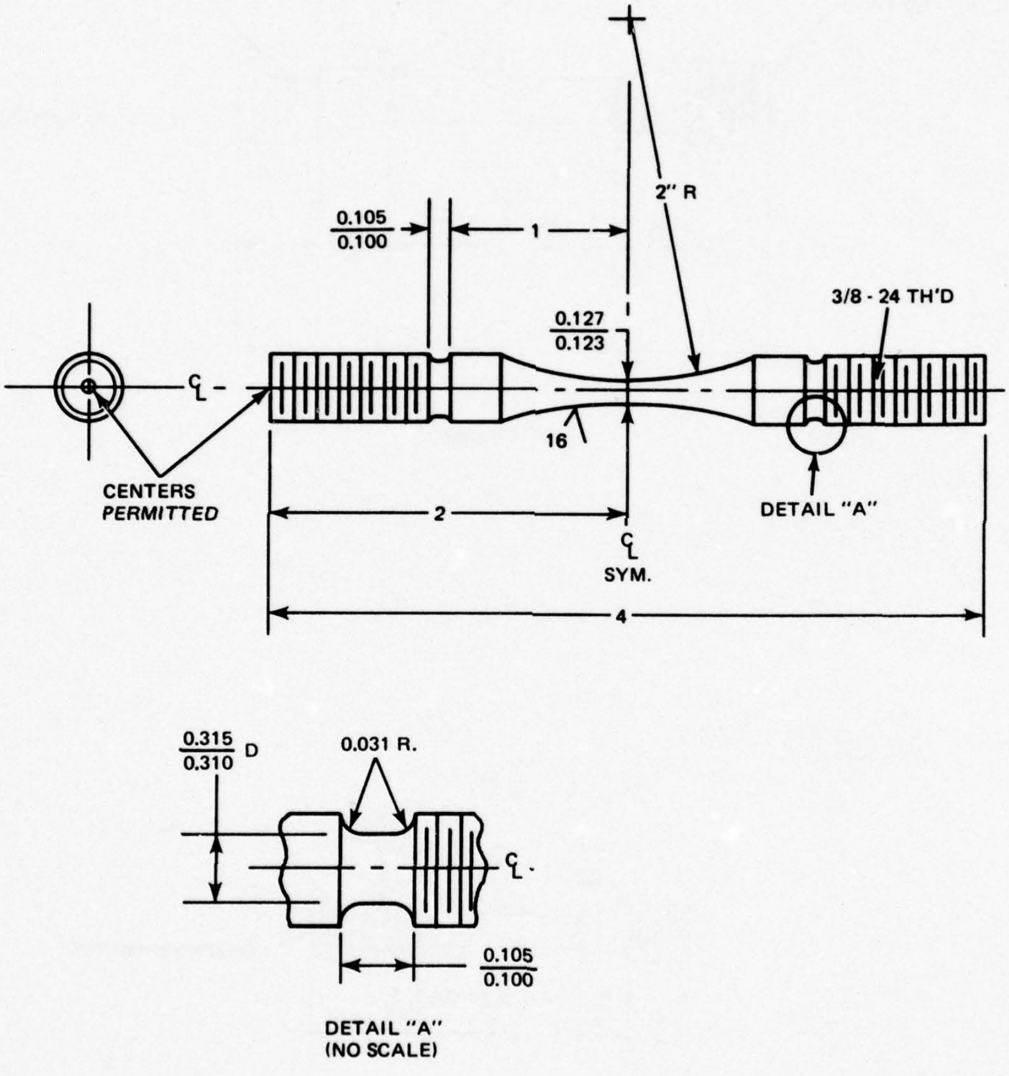
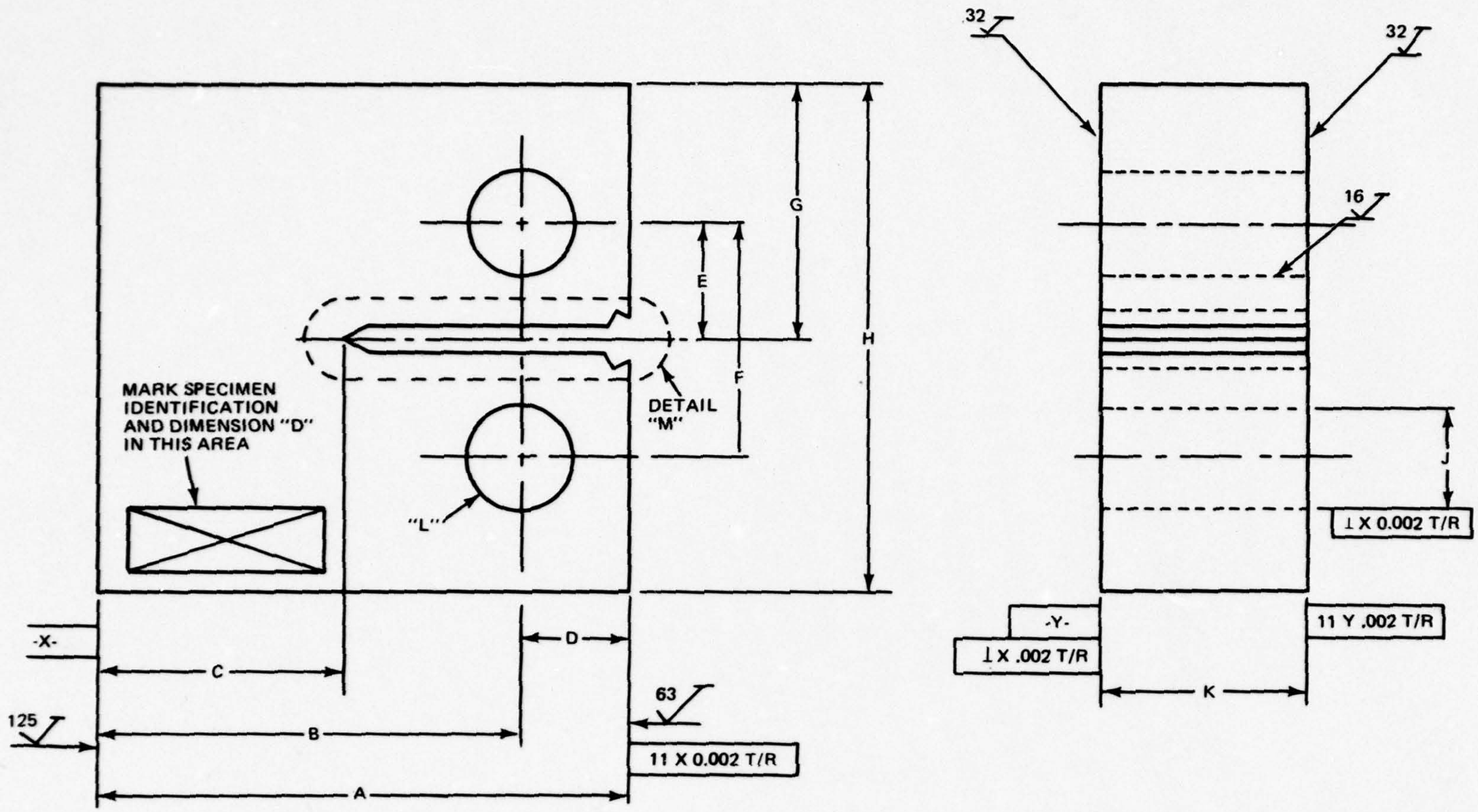


Figure 34. Fatigue Specimen Configuration (45 MPS 196)



DIMENSIONAL CLASSIFICATION									
DESIG.	A ±0.005	B ± 0.002	C ± 0.010	E ± 0.002	F ± 0.005	G ± 0.005	H ± 0.005	J +0.002 -0.000	K ± 0.010
K_{IC}	3.125	2.500	1.475	0.687	1.375	1.500	3.000	0.625	1.250
K_g	1.25	1.000	0.650	0.275	0.500	0.600	1.200	0.250	0.500

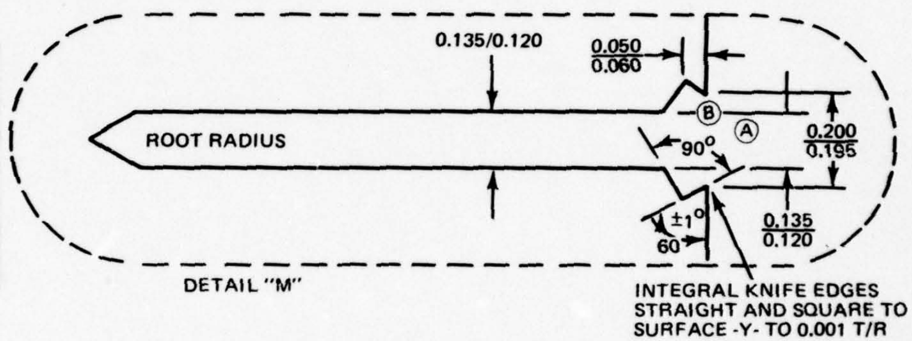


Figure 35. Compact Tension Specimen for K_{IC} (Fracture Toughness) With Integral Knife Edges – TGS 1094

Appendix C

PHASE 2 BRACE DIMENSIONAL ANALYSIS

Table XV Dimensional Comparison of Braces from Run 1 (Task 1)

Dimension No.	Target (inches)	SM-404 (inches)	SM-405 (inches)
1	11.250	11.250	11.250
2	10.050	9.980	9.980
3	6.950	6.950	6.950
4	4.781	4.788	4.788
5	3.438	3.438	3.438
6	1.375	1.436	1.436
7	0.100	0.097/0.143	0.097/0.143
8	0.125	0.134/0.137	0.134/0.137
9	15°36'	15°36'	15°36'
10	0.190	0.226/0.233	0.226/0.233
11	2.000	2.000	2.000
12	0.800	0.800	0.800
13	0.200	0.186/0.210	0.186/0.210
14	0.38R	0.38R	0.38R
15	0.250	0.271/0.275	0.271/0.275
16	0.100	0.154/0.165	0.154/0.165
17	22°42'	22°42'	22°42'
18	1.375	1.436	1.436
19	1.438	1.438	1.438
20	2.563	2.563	2.563
21	0.200	0.290	0.290
22	4°38'	4°38'	4°38'
23	0.344	0.330	0.330
24	0.190	0.185/0.210	0.185/0.210
25	0.090	0.065/0.090	0.065/0.090
26	0.320	0.360	0.360
27	1.525	1.495	1.495
28	3.140	3.095/3.115	3.095/3.115
29	0.700	0.680	0.680
30	1.500	1.490	1.490
31	0.100	0.120	0.120
32	0.050	0.055	0.055
33	0.200	0.225/0.250	0.225/0.250
34	0.950	0.930	0.930
35	2.000	1.985	1.985
36	3.408	3.315	3.315
37	0.030	0.123	0.123
38	7.130	7.120	7.120
39	0.055	0.035	0.035
40	1.200	1.270	1.270

Table XVI Dimensional Comparison of Braces from Run 3 (Task 1)

Dimension No.	Target (inches)	SM-446 (inches)	SM-447 (inches)
1	11.250	11.278	11.258
2	10.050	9.993	9.958
3	6.950	7.011	6.918
4	4.781	4.803	4.788
5	3.438	3.438	3.438
6	1.375	1.493	1.503
7	0.225	0.174/0.212	0.177/0.213
8	0.125	0.128/0.139	0.133/0.145
9	15°35'	13°44'	13°44'
10	0.315	0.279/0.325	0.265/0.325
11	2.245	2.200	2.120/2.200
12	1.045	1.100	1.100
13	0.325	0.248/0.316	0.263/0.320
14	0.38R	0.38R	0.38R
15	0.375	0.321/0.344	0.348/0.356
16	0.225	0.275/0.290	0.275/0.300
17	22°42'	22°22'	22°
18	1.323	1.493	1.513
19	1.303	1.440	1.466
20	2.813	2.835	2.860
21	0.325	0.440	0.440
22	4°38'	5°18'	5°24'
23	0.375	0.360	0.360
24	0.190	0.175/0.182	0.175/0.180
25	0.090	0.100	0.090
26	0.320	0.365	0.350
27	1.515	1.490	1.500
28	3.140	3.080	3.085
29	0.700	0.680	0.690
30	1.500	1.460	1.465
31	0.100	0.100	0.094
32	0.050	0.066	0.060
33	0.200	0.210/0.225	0.218/0.250
34	0.950	0.930	0.930
35	2.000	≥ 1.975	1.975/1.995
36	3.408	3.378	3.298
37	0.030	0.060	0.060
38	7.130	7.158	7.178
39	0.055	0.060	0.060
40	1.200	1.295	1.285

Table XVII Dimensional Comparison of Braces from Run 4 (Task 1)

Dimension* No.	Target (inches)	SM-487 (inches)	SM-488 (inches)
1	11.250	11.258	11.278
2	10.050	9.958	9.978
3	6.950	6.918	6.943
4	4.781	4.788	4.783
5	3.438	3.438	3.438
6	1.375	1.503	1.478
7	0.225	0.188/0.205	0.182/0.210
8	0.125	0.118/0.131	0.120/0.130
9	15°35'	13°34'	13°45'
10	0.315	0.258/0.324	0.267/0.328
11	2.245	2.200	2.210
12	1.045	1.100	1.110
13	0.325	0.270/0.285	0.280/0.297
14	0.38R	0.38R	0.38R
15	0.375	0.367	0.363
16	0.225	0.281/0.300	0.260/0.270
17	22°42'	23°23'	22°25'
18	1.323	1.513	1.478
19	1.303	1.460	1.430
20	2.813	2.875	2.825
21	0.325	0.440	
22	4°38'	5°15'/5°30'	5°14'
23	0.375	0.360	0.360
24	0.190	0.165/0.181	0.170/0.177
25	0.090	0.075	0.080
26	0.320	0.350	0.340
27	1.515	1.518	1.518
28	3.140	3.125	3.125
29	0.700	0.700	0.700
30	1.500	1.490	1.495
31	0.100	0.090	0.090
32	0.050	0.050	0.045
33	0.200	0.200/0.210	0.195/0.215
34	0.950	0.950	0.945
35	2.000	2.010	2.015
36	3.408	3.298	3.298
37	0.030	0.140	
38	7.130	7.178	7.178
39	0.055	0.060	0.060
40	1.200	1.300	1.300

*Appendix A, Figure 31, shows part drawing with dimension locations.

Table XVIII Dimensional Comparison of Braces from Run 5 (Pilot Lot)

Dimension* No.	Target (inches)	SM-506 (inches)	SM-507 (inches)	SM-513 (inches)	SM-514 (inches)	SM-515 (inches)
1	11.250	11.163	11.198	11.250	11.270	11.219
2	10.050	9.908	9.937	9.940	9.938	9.919
3	6.950	6.899	6.925	6.914	6.931	6.902
4	4.781	4.760	4.776	4.776	4.781	4.774
5	3.438	3.438	3.438	3.438	3.438	3.438
6	1.375	1.480	1.462	1.458	1.453	1.467
7	0.225	0.160/0.192	0.158/0.190	0.165/0.194	0.144/0.175	0.13[illegible]/0.171
8	0.125	0.126/0.130	0.124/0.135	0.130/0.135	0.126/0.129	0.127/0.132
9	15°35'	15°35'	15°35'	15°35'	15°35'	15°35'
10	0.315	0.256/0.273	0.267/0.287	0.225/0.228	0.240/0.255	0.250/0.262
11	2.245	2.150	2.154	2.160	2.130	2.135
12	1.045	0.925	0.924	0.900	0.884	0.910
13	0.325	0.254/0.286	0.251/0.271	0.271/0.315	0.245/0.273	0.267/0.287
14	0.38R	0.38R	0.38R	0.38R	0.38R	0.38R
15	0.375	0.332/0.340	0.327/0.329	0.357/0.364	0.316/0.332	0.343/0.351
16	0.225	0.275/0.285	0.271/0.285	0.277/0.295	0.270/0.287	0.270/0.285
17	22°42'	22°42'	22°42'	22°42'	22°42'	22°42'
18	1.323	1.480	1.462	1.458	1.453	1.467
19	1.303	1.400	1.390	1.380	1.408	1.410
20	2.813	4.813	2.723	2.730	2.707	2.705
21	0.325	0.390	0.406		0.406	0.390
22	4°38'	4°38'	4°38'	4°38'	4°38'	4°38'
23	0.375		0.395	0.385	0.390	0.415
24	0.190	0.255	0.255	0.215	0.215	0.227/0.232
25	0.090	0.210	0.215	0.215	0.215	0.195
26	0.320		0.290	0.320	0.320	0.330
27	1.515	1.505	1.510	1.505	1.525	1.502
28	3.140	3.105	3.120	3.100	3.110	3.105
29	0.700	0.690	0.700	0.695	0.715	0.708
30	1.500	1.500	1.490	1.480	1.490	1.495
31	0.100	0.070/0.090	0.070/0.090	0.070/0.090	0.070/0.090	0.070/0.090
32	0.050		0.055/0.060	0.056	0.060/0.075	
33	0.200		0.210	0.202/0.212	0.200/0.208	0.207/0.215
34	0.950		0.960	0.950	0.960	
35	2.000	2.010/2.030	2.030	2.000	2.000	2.000/2.015
36	3.408	3.408	3.408	3.408	3.408	3.408
37	0.030					
38	7.130	7.143	7.185	7.200	7.216	7.203
39	0.055		0.131	0.157	0.196	
40	1.200	1.255	1.261	1.310	1.332	1.300

*Appendix A, Figure 31, shows part drawing with dimension locations.

Table XVIII Dimensional Comparison of Braces from Run 5 (Pilot Lot) (Cont)

DIMENSION NO.	TARGET (inches)	SM-508 (inches)	SM-509 (inches)	SM-510 (inches)	SM-511 (inches)
1	11.250	11.234	11.229	11.230	11.249
2	10.050	9.951	9.647	9.938	9.973
3	6.950	6.913	6.932	6.919	6.903
4	4.781	4.785	4.772	4.778	4.766
5	3.438	3.438	3.438	3.438	3.438
6	1.375	–	–	–	–
7	0.225	0.143/0.151	0.151/0.171	0.175/0.180	0.088/0.100
8	0.125	0.125/0.129	0.128/0.135	0.123/0.128	0.125/0.130
9	15°35′	–	–	–	–
10	0.315	0.251/0.259	0.274/0.276	0.281/0.284	0.178/0.189
11	2.245	2.120	2.175	2.175	1.998
12	1.045	0.911	1.045	0.956	0.788
13	0.325	0.280/0.282	0.304/0.306	0.292/0.302	0.198/0.207
14	0.38R	–	–	–	–
15	0.375	0.309/0.319	0.358/0.362	0.348/0.349	0.240/0.251
16	0.225	0.262/0.283	0.282/0.290	0.180/0.186	0.098/0.107
17	22°42′	–	–	–	–
18	1.323	–	1.491	1.485	1.573
19	1.303	1.344	1.332	1.353	1.469
20	2.813	2.672	2.724	2.742	2.545
21	0.325	0.422	0.426	0.431	0.220
22	4°38′	–	–	–	–
23	0.375	–	–	–	–
24	0.190	–	–	–	–
25	0.090	0.145	0.150	0.156	0.102
26	0.320	–	–	–	–
27	1.515	1.503	1.500	1.500	1.517
28	3.140	3.105	3.103	3.105	3.120
29	0.700	0.684	0.702	0.696	0.695
30	1.500	1.490	1.496	1.495	1.500
31	0.100	0.080/0.090	0.075/0.090	0.071/0.084	0.074/0.087
32	0.050	–	–	–	–
33	0.200	–	–	–	–
34	0.950	–	–	–	–
35	2.00	2.00/2.025	1.990/2.020	2.003/2.025	2.003/2.011
36	3.408	3.312	3.429	3.404	3.337
37	0.030	–	–	–	–
38	7.130	7.170	7.185	7.182	7.122
39	0.055	–	–	–	–
40	1.200	1.281	1.280	1.292	–

Appendix D

FATIGUE AND TENSILE DATA FOR RUN 5

Table XIX Tensile Test Data for Runs 1, 2, 4 and 5*

HIP Run	S/N	Spec	Test Config.	Heat Treatment	Ftu, ksi	Fty, ksi	Elong, %	RA, %
1	402	1	TGS 5307	None	148.8	142.7	17	34.7
		2	"	"	149.9	142.5	18	37.3
		6	5308	None	149.7	142.9	18	35.8
		7	"	"	148.2	140.9	18	32.6
		30	5307	1300°F/2 hr	148.0	141.0	18	43
2	432	23	5307	None	146.7	135.7	15	44
		24	"	"	142.8	133.6	14.5	43.8
		51	"	1300°F/24 hr	142.5	135.1	22	47.8
4	485	53	5307	1300°F/24 hr	144.2	136.4	20	42.2
5	502	69	5307	1300°F/24 hr	145.5	137.4	17	32.8
		70	"	"	145.5	137.6	16.5	30.8
		71	5303	"	150.2	141.2	16	40.4
		72	"	"	150.2	140.1	17	44.1

* Results from Run 5 before re-HIP.

Table XX Fatigue Endurance Data

HIP Run	S/N	Spec No.	TGS Test Config.	Treatment	Stress, ksi	Life, Cycles X10^3
1	402	3	5771	None	100	540
		4	"	"	85	160
		5	"	"	85	180
		10	45 MPS 196	"	80	3,900
		11	"	"	75	5,700
		12	"	"	100	804
	403	20	5771	"	110	30
2	432	22	5771	1300°F/24 hr +chem-mill (0.005 inch) + shot peen	90	442
		19	45 MPS 196	1300°F/16 hr	80	2,800
		35	"	1300°F/24 hr	80	13,800 N.F.
					R110	35
		36	"	1450°F/16 hr	80	70
		41	"	1300°F/24 hr	90	5,200
		42	"	1300°F/2 hr	90	500
		43	"	1300°F/24 hr	85	2,500
4	485	45	5771	1300°F/24 hr	85	600
		46	"	1300°F/2 hr	90	126
		47	45 MPS 196	1300°F/24 hr	90	2,400
		48	"	1300°F/24 hr	85	2,100

Table XX Fatigue Endurance Data (Cont'd)

HIP Run	S/N	Spec No.	TGS Test Config.	Treatment	Stress, ksi	Life, Cycles X10^3
5	499	55	5771	1300°F/24 hr	75	7,260
		56	"	"	80	92
		57	"	"	90	63
		58	"	"	90	63
		59	"	"	100	59
		60	"	"	100	44
		73	5771	1300°F/2 hr FC to 800°F	AC90	324
		74	"	"	90	95
		75	"	800°F/6 hr	90	30
		76	"	"	90	116
	502	62	5771-3	1300°/24 hr Kt=3.3	35	108
		65	"	"	42	13
		64	"	"	25	134
	505	83	5771	1650°F/1 hr FC to 1300°F/2 hr	90	69
		84	"	"	90	84
		85	"	1450°F/2 hr FC to 1300°F/2 hr	90	70
		85	"		90	88
		89	"	1300°F/2 hr	90	222
		90	"	"	90	164

APPENDIX E

PROPOSED AEROSPACE SPECIFICATIONS

E-1

PROPOSED AEROSPACE MATERIAL SPECIFICATION
TITANIUM NEAR-NET SHAPE COMPONENTS, ANNEALED, FOR AIRFRAME APPLICATIONS
ALLOY Ti-6Al-6V-2Sn

1. SCOPE

1.1 Scope

This specification covers a titanium alloy powder metallurgy product in the form of near-net shapes intended for structural airframe applications.

2. APPLICABLE DOCUMENTS

SPECIFICATIONS

Aerospace Material Specification

AMS 2249	Chemical Check Analysis Limits, Titanium and Titanium Alloys
AMS 2350	Standards and Test Methods

STANDARDS

Federal

Federal Test Method Standard No. 151	Metals, Test Methods

Military

MIL-STD-163	Steel Mill Products, Preparation for Shipment and Storage
MIL-I-6866	Inspection, Penetrant Method of
MIL-STD-453	Inspection, Radiographic

OTHER PUBLICATIONS

American National Standards Institute, 1430 Broadway, New York, N. Y. 10018

ANSI B46.1	Surface Texture

American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103

ASTM E120	Chemical Analysis of Titanium and Titanium-Base Alloys

3. REQUIREMENTS

3.1 General Material Requirements

Shapes shall be produced by consolidation of spherical Ti-6Al-6V-2Sn powder meeting *Grumman Specification*, by hot isostatic pressing to produce a product meeting the requirements specified herein.

3.1.1 Quality

The product, as received by purchaser, shall be uniform in quality and condition, sound, and free from foreign materials and from internal and external imperfections detrimental to usage of the product.

3.1.2 Product Characteristics

3.1.2.1 Tolerances. Non-machined surfaces shall be as specified on the applicable drawing. Surfaces to be machined shall be prepared by the vendor such that the removal of a specified amount of material from each applicable surface will render all such surfaces conformable to the applicable drawing.

3.1.2.2 Surface Finish. The surfaces of shapes shall be equivalent to 125 microinches (3.2 mm) or better determined in accordance with ANSI B46.1.

3.1.2.3 Density. Shapes shall be 100% dense as determined by metallographic examination of pour spouts and/or prolongations. One part per lot shall be destructively inspected and tested per 3.1.2.5.

3.1.2.4 Heat Treatment. All shapes, with consolidation container removed, shall be vacuum annealed by heating to 1300°F±25 (704°C±14) at 10^{-4} microns of Hg or better, holding at heat for 2 hr ±0.5, and cooling in vacuum.

3.1.2.5 Mechanical Properties. A shape after heat treatment as in 3.1.2.4 shall meet the following tensile property requirements, determined in accordance with ASTM E8:

	Class A (Critical Parts)	Class B (Non-Critical Parts and Forging Preforms)
Tensile Strength, min	150,000 psi	140,000 psi (965 MPa)
Yield Strength at 0.2% Offset	140,000 psi	130,000 psi (909 MPa)
Elongation in 2 in. (50.8 mm) or 4D, min	8%	15%
Reduction of Area, min	20%	30%

3.1.2.6 Composition. Shall conform to the following percentages by weight, determined by wet chemical methods in accordance with ASTM E120, by spectrographic methods in accordance with Federal Test Method Standard No. 151, Method 112, except that oxygen and hydrogen shall be determined by a vacuum fusion method, or by other approved analytical methods; hydrogen shall be determined before and after vacuum heat treatment:

Element	Wt. %
Aluminum	5.00 - 6.00
Vanadium	5.00 - 6.00
Iron	0.35 - 1.00
Carbon	0.05 (Max.)
Nitrogen	0.04 (Max.)
Oxygen	0.20 (Max.)
Hydrogen	0.015 (Max.)
Copper	0.34 - 1.00
Other	0.40 (Max.)
Tin	1.50 - 2.50
Titanium	Balance

3.1.2.6.1. Check Analysis. Composition variations shall meet the requirements of AMS 2249.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection

Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the purchaser. The purchaser reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.1.1 Qualification

Laboratories and personnel performing penetrant and radiographic inspections shall be qualified in accordance with MIL-I-6866, and MIL-STD-453, respectively.

4.2 Sampling Requirements

Type of tests to be conducted and required number of test specimens for each production lot will be specified on the purchase order and will be based on confidence data established for each vendor. A production lot includes all configurations consolidated from an identical lot of powder and processed in the same autoclave run.

4.3 Tests

4.3.1 Visual Inspection

Each configuration in the production lot will be visually inspected at 10x magnification for presence of surface porosity, cracks and pits. Parts exhibiting these defects will be rejected.

4.3.2 Penetrant Inspection

Each configuration in the production lot will be inspected in accordance with MIL-I-6866. Presence of defects listed in 4.3.1 will lead to the rejection of the part.

4.3.3 Radiographic Inspection

Configurations to be inspected by radiographic techniques will be specified in the purchase order. Parts with porosity indications in excess of limits for wrought counterparts will be subject to an outright rejection. In addition, all surface-connected porosity

is rejectable, regardless of size. Presence of inclusions, regardless of size, will also be a cause for rejection. Questionable cases will be resolved using the Image Enhancement System.

4.3.4 Metallographic Evaluations

Sections of pour spouts, prolongations and/or auxiliary test blocks will be submitted for metallographic evaluations as specified in the purchase order. Detection of inclusions or bulk microporosity on polished and etched surfaces at 100x magnification will be a cause for rejection of the entire production lot.

4.3.5 Dimensional Analysis

Selected configurations from each production lot will be subjected to a dimensional analysis, as specified in the purchase order to ascertain that obtainable dimensions remain within specified tolerances.

4.3 Approval

4.3.1 Shape Consolidation

Preproduction consolidation of powder into shapes shall be approved by purchaser before shapes for production use are supplied. Approval of shapes shall in no way relieve the shape vendor of responsibility for continued performance to all purchase order requirements.

4.3.2 HIP Practice

Limits and controls shall be established for producing the finished part. Once effectiveness is demonstrated by first acceptance, the HIP procedures and controls shall not be changed without prior approval from Grumman Materials and Processes.

4.3.2.1 <u>Process Control Procedures</u>

A summary of the processes and controls used to produce shapes shall be prepared and submitted by the HIP supplier for Grumman approval. This summary shall include raw material acceptance criteria, canning method, vacuum degas procedure, autoclave loading, temperature monitoring system, thermal and pressure history, number of parts per lot, surface conditioning, heat treatment temperature and times, and intermediate and final inspection procedures. Changes to previously approved procedures shall, at the discretion of Grumman Materials and Processes, require a new first-article qualification prior to final approval. Minor changes (within the scope of this specification) to the

approved process control procedure, on lot basis, such as autoclave loading pattern, will require a Grumman/Seller Quality Surveillance Report (SQSR) and shall be so noted on the Certificate of Conformance for the subject lot.

4.4 Reports

The vendor of the product shall furnish with each shipment three copies of a report showing the results of tests to determine conformance to the acceptance test requirements and a statement that the product conforms to the other technical requirements of this specification. This report shall include the purchase order number, material specification number, lot number, part number of shape, quantity, and the powder lot number, size, and source of powder used to make the shapes.

5. PREPARATION FOR DELIVERY

5.1 Identification

Shall be as agreed upon by purchaser and vendor, except that the identification shall include not less than the following information:

TITANIUM ALLOY SHAPE, 6Al-6V-2Sn

MANUFACTURER'S IDENTIFICATION ____________________

PURCHASE ORDER NUMBER ____________________

SIZE OR PART ____________________

QUANTITY ____________________

LOT NUMBER ____________________

5.2 Packaging

The product shall be prepared for shipment in accordance with commercial practice to assure carrier acceptance and safe transportation to the point of delivery. Packaging shall conform to carrier rules and regulations applicable to the mode of transportation.

6. ACKNOWLEDGEMENT

A vendor shall mention this specification number in all quotations and when acknowledging purchaser orders.

7. REJECTIONS

Material not conforming to this specification or to authorized modifications will be subject to rejection.

E-2

PROPOSED AEROSPACE MATERIAL SPECIFICATION FOR Ti-6Al-6V-2Sn TITANIUM ALLOY POWDER

1. SCOPE

1.1 Scope

This specification covers a titanium alloy in the form of prealloyed powder primarily intended for consolidation into near-net shapes for use in noncritical airframe applications.

2. APPLICABLE DOCUMENTS

Aerospace Material Specifications

AMS 2249	Chemical Check Analysis Limits, Titanium and Titanium Alloys
AMS 2350	Standards and Test Methods
AMS 2380	Approval and Control of Premium-Quality Titanium Alloys
AMS 2635	Radiographic Inspection

Federal Standards

Federal Test Method Standard No. 151	Metals; Test Methods

Military Standards

MIL-STD-794	Parts and Equipment, Procedure for Packaging and Packing of
ASTM Publications	Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa 19103.
ASTM B214	Sieve Analysis for Granular Metal Powders
ASTM B243	Powder Metallurgy, Definition of Terms Used in
ASTM E120	Chemical Analysis of Titanium and Titanium-Base Alloys

3. REQUIREMENTS

3.1 General Material Requirements

Shall be a prealloyed spherical powder, essentially free of splat and large agglomerated masses, produced in an inert atmosphere or vacuum by a fusion process from ingots alloyed from material in accordance with the raw material control provisions of AMS 2380. The powder shall conform to the composition requirements of 3.1.2.1 and shall have size distribution as shown in 3.1.2.2.

3.1.1 Quality

3.1.1.1 Raw Material. Ingot from which powder is made shall be produced in accordance with AMS 2380 using triple melting techniques, with the final melting occurring during powder production when a fusion method is used to produce powder. With prior approval of the purchaser, the second melting may use non-consumable electrode techniques to cast consumable electrodes for the third melting during powder production, provided a fusion technique is used.

3.1.1.2 Powder. The powder, as received by purchaser, shall be uniform in color and quality, dry, and free from foreign materials and imperfections detrimental to its performance during consolidation or in resultant net shapes.

3.1.2 Product Characteristics

3.1.2.1 Composition. Shall conform to the following percentages by weight, determined by wet chemical methods in accordance with ASTM E120, by spectrographic methods in accordance with Federal Test Method Standard No. 151, Method 112, except that oxygen and nitrogen shall be determined by a vacuum fusion method, or by other approved analytical methods:

Element	Wt. %
Aluminum	5.00 - 6.00
Vanadium	5.00 - 6.00
Iron	0.35 - 1.00
Carbon	0.05 (Max.)
Nitrogen	0.04 (Max.)
Oxygen	0.20 (Max.)
Hydrogen	0.015 (Max.)
Copper	0.35 - 1.00

Element	Wt. %
Other	0.40 (Max.)
Tin	1.50 - 2.50
Titanium	Balance

3.1.2.1 Check Analysis. Composition variations shall meet the requirements of AMS 2249.

3.1.2.2 Particle Size Distribution. Not less than 75% of the particles by weight shall pass through the No. 45 (354 microns) sieve while being retained by the No. 170 (88 microns) sieve. In the bulk analysis all of the particles shall pass through the No. 35 (500 microns) sieve, and not more than 5% by weight shall pass through the No. 325 (45 microns) sieve.

3.1.2.3 Powder Consolidation and Evaluation. A randomly selected sample of approximately 0.75 - 1.0 lb (340 - 450 g) from each powder lot shall be hot consolidated using a method which will not contaminate the powder particles during consolidation. Each consolidated sample having a theoretical density of not less than 98.5 percent shall be divided into panels of not less than 18 sq. in. (11, 600 mm^2) area with thickness of 0.200 in., +0.015, -0.025 (5.08 mm, +0.38, -0.64). Consolidated panels shall contain no high-density inclusions larger than 0.050 inch, or low-density inclusions larger than 0.005 inch, determined by radiographic examination in accordance with AMS 2635. The determination of oxygen content shall be performed on these panels and reported for the lot represented.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection

The vendor of the powders shall supply all samples and shall be responsible for performing all required tests. Results of such tests shall be reported to the purchaser as required in 4.5. Purchaser reserves the right to perform such confirmatory testing as he deems necessary to assure that the powder conforms to the requirements of this specification.

4.2 Classification of Tests

Tests to determine conformance to all technical requirements of this specification are classified as acceptance or routine control tests.

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FLIGHT QUALIFICATION OF TITANIUM F-14A AIRFRAME COMPONENTS MANU--ETC(U)

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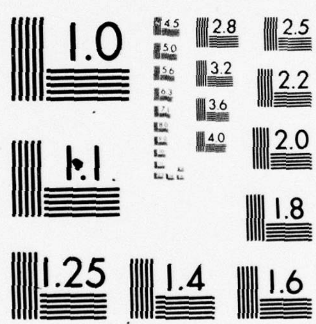

MICROCOPY RESOLUTION TEST CHART

NATIONAL BUREAU OF STANDARDS-1963-A

4.3 Sampling

Sufficient material shall be taken at random from each lot to perform each test in duplicate. Sampling for oxygen determination shall be from panels prepared in accordance with 3.1.2.3.

4.3.1 Lot. A lot shall be all powder produced from common feed material (an ingot, billet, or case electrode from a common ingot) in one powder production run of the equipment. When approved by purchaser, a lot may consist of the powder produced from one ingot, or the billets from a common ingot, produced in a consecutive series of runs in the same equipment and under the same fixed parametric conditions with the powder thoroughly blended prior to sampling.

4.4 Approval

4.4.1 Sample powder shall be approved by purchaser before powder for production use is supplied, unless such approval be waived.

4.4.2 Vendor shall use materials, processing techniques, and methods of routine inspection on production powder which are essentially the same as those used on the approved sample powder. If any change is necessary in ingredients, in processing techniques, or in methods of routine inspection, vendor shall submit for reapproval a statement of the proposed changes in materials and processing and, when requested, sample powder produced by the revised process. No production powder made by the revised procedure shall be shipped prior to receipt of re-approval.

4.5 Reports

4.5.1 The vendor of the powder shall furnish with each shipment three copies of a report of the results of tests for the chemical composition of each heat and the oxygen content and particle size distribution of each lot in the shipment and a statement that the powder conforms to the other technical requirements of this specification. This report shall include the purchase order number, material specification number, vendor's product designation, feed material, lot number, and quantity.

4.5.2 When parts requiring use of this powder are supplied, the vendor of finished or semi-finished parts shall furnish with each shipment three copies of a report showing the purchase

order number, this specification number, lot number, contractor or direct supplier or powder, part number, and quantity. When powder for making parts is produced or purchased by the parts vendor, that vendor shall inspect each lot of powder to determine conformance to the requirements of this specification, and shall include in the report a statement that the powder conforms, or shall include copies of laboratory reports showing the results of tests to determine conformance.

5. PREPARATION FOR DELIVERY

5.1 Packaging and Identification

5.1.1 The powder shall be packaged in individual containers of the size ordered or the size required to assure acceptance and safe transport to the point of delivery. A lot may be packaged in smaller quantities and delivered separately under the basic lot approval as long as lot identity is maintained. Each container shall be thoroughly cleansed and dried immediately prior to filling, and shall be sealed immediately after filling to protect the contents from contamination during shipment and during storage under normal dry storage conditions. Seals used on the containers shall be so designed that they must be destroyed in order for the container to be opened.

5.1.2 Each individual container shall be permanently and legibly marked to give not less than the following information:

TITANIUM ALLOY POWDER, 6Al-6V-2Sn

GRUMMAN SPECIFICATION NO.

MANUFACTURER'S IDENTIFICATION ______________________

PURCHASE ORDER NUMBER ______________________

QUANTITY ______________________

LOT NUMBER ______________________

5.1.3 Individual containers may be packaged in an exterior shipping container capable of protecting the materials during transit and storage against damage from exposure to weather or any normal hazard.

5.1.4 Each exterior shipping container shall be legibly marked with not less than the following information in such a manner that the markings will not smear or be obliterated during normal handling and use:

TITANIUM ALLOY POWDER, 6Al-6V-2Sn

GRUMMAN SPECIFICATION NO.

MANUFACTURER'S IDENTIFICATION ______________________

PURCHASE ORDER NUMBER ______________________

QUANTITY ______________________

LOT NUMBER ______________________

SPECIAL MARKINGS (as applicable)

5.1.5 Containers shall be prepared for shipment in accordance with commercial practice to assure carrier acceptance and safe transportation to the point of delivery and in compliance with applicable regulations pertaining to the packaging and handling of this material. Packaging shall conform to carrier rules and regulations applicable to the mode of transportation.

6. ACKNOWLEDGEMENT

A vendor shall mention this specification number in all quotations and when acknowledging purchase orders.

7. REJECTIONS

Powder not conforming to this specification or to authorized modifications will be subject to rejection.

8. NOTES

8.1 Definitions

For definitions of terms used herein, refer to ASTM B243.

E-3

PROPOSED AEROSPACE MATERIAL SPECIFICATION

HOT ISOSTATIC PRESSING (HIP) OF TITANIUM ALLOY POWDER

1. SCOPE

1.1 Scope

This specification presents requirements for hot isostatic pressing of titanium alloy powder forging preforms and HIP parts.

1.1.1 Classification: This specification contains the following classes:

Class A - HIP parts

Class B - Forging Preforms

The requirements specified herein apply to all classes unless otherwise specified.

1.2 Definitions: For purposes of this specification, the following definitions shall apply:

Part Lot - Parts produced in the same autoclave cycle and from same heat of powder.

Purchaser - The procurement activity that issued the procurement document invoking this specification.

Working Zone - The volume of the heated region of an autoclave which may be occupied by parts or material to be hot isostatically pressed.

2. APPLICABLE DOCUMENTS

2.1 The following documents shall form a part of this specification to the extent specified herein. Unless a specific issue is specified, the latest revision shall apply.

American Society for Testing and Materials

ASTM E230 Temperature-Electromotive Force (EMF) Tables for Thermocouples

3. EQUIPMENT

3.1 Autoclave

3.1.1 Autoclaves shall be of the inert gas pressurization type, internally heated, cold wall pressure vessel.

3.2 Fixtures

3.2.1 Suitable jigs, trays, baskets, hangers, racks or other fixtures shall be provided as necessary for proper handling and positioning of materials to be hot isostatic pressed. All fixtures shall be made of material which is compatible with the material to be treated, or adequately isolated to assure that undesirable reactions or mechanical distortion do not occur.

3.3 Containers

3.3.1 Powder containers shall be made from materials demonstrated to have no adverse effect on the titanium.

3.4 Temperature Measurement and Control Devices

3.4.1 Temperature Measurement

3.4.1.1 Temperature measuring and recording devices shall be provided for the autoclave. The devices shall be of the potentiometric type, shall use thermocouple sensors, and shall provide permanent records of the temperature during the entire treatment.

3.4.2 Temperature Control

3.4.2.1 A sufficient number of suitable automatic temperature control devices shall be provided and properly arranged in the autoclave to assure the required temperature control in the working zone. The devices shall be of the potentiometric type and shall use thermocouple sensors.

3.5 Pressure Measurement Devices

3.5.1 Pressure measurement devices shall be accurate to within + or - two percent at the maximum operating pressure and shall be capable of continuously monitoring and recording the pressure in the autoclave throughout the process.

4. PROCEDURE

4.1 General

4.1.1 All processing equipment and significant processing parameters shall be approved by the Purchaser. Once a technique for producing a specific part or preform has been established and approved, no changes shall be made without prior approval of the Purchaser.

4.1.2 All hot isostatic pressing facilities shall be qualified in accordance with Section 5 of this specification prior to production.

4.2 Cleaning

4.2.1 All containers and fixtures shall be properly cleaned to remove all loose particles and all surface contaminants which may be detrimental to the material being treated or to autoclave components.

4.3 Container Filling

4.3.1 Leak Check: All HIP containers shall be leak tested by an appropriate procedure.

4.3.2 Filling and Sealing: Powder or powder-filled molds shall be loaded into the container and the loaded container outgassed and sealed by a method approved by the Purchaser.

4.4 Loading of Autoclave

4.4.1 All material to be treated shall be located within the working zone in such a manner as to facilitate pressurization of the chamber and assure uniform heating and cooling of the material.

4.5 Instrumentation

4.5.1 A minimum of three thermocouples shall accompany the material during treatment. They shall be located in the hottest, coldest and nominal temperature regions of the working zone which is in use as determined by the temperature uniformity qualification test. The thermocouples shall be in close proximity to the containers. An alternate instrumentation plan may be used with prior approval of the Purchaser.

4.6 Time, Temperature and Pressure

4.6.1 The autoclave heat-up, pressurization and cooling cycles shall be approved by the Purchaser.

4.7 Pressure Environment

4.7.1 Equipment: All pressure indicating equipment shall be adjusted in accordance with the instrument manufacturer's instructions.

4.7.2 Pressure: The chamber pressure during treatment shall be 15,000 psi minimum for two hours minimum at temperature. During heat-up, treatment, and cool down, the chamber pressure shall be continuously recorded. The specific pressure/time conditions shall be subject to approval by the Purchaser.

4.8 Thermal Treatment

4.8.1 The times and temperatures for the thermal cycle shall be as agreed upon between Purchaser and vendor. The temperatures shall be continuously measured and recorded with respect to time during the entire thermal cycle. The use of multi-point recorders with a periodic printout of five minutes maximum per thermocouple is permitted.

4.9 Density

4.9.1 Class A: HIP parts shall have a density greater than 99.8 percent of a HIP test sample fabricated from the same powder heat or master powder blend. The powder pressing and forging processes used on the test sample shall be by methods agreed upon by the Purchaser and the vendor.

Class B: As-compacted preforms shall have a density greater than 99.6 percent of a compacted plus forged test sample fabricated from the same powder heat or master powder blend. The powder compaction and forging processes used on the test sample shall be by methods agreed upon by the Purchaser and the vendor.

4.10 Microstructure

4.10.1 HIP parts or preforms shall have a uniform microstructure with no outlining of prior particle boundaries or evidence of voids.

4.11 Decanning

4.11.1 HIP containers shall be completely removed by a method approved by the Purchaser.

4.12 Re-HIP

4.12.1 Re-HIP procedures, when permitted, shall be as defined in a Quality Control Plan approved by the Purchaser.

4.13 Inspections and Tests

4.13.1 All inspections or tests required by the drawing or applicable specifications shall be performed. The results of these inspections or tests shall meet the requirements of the drawing or applicable specifications.

4.13.2 Sample parts or representative material shall be evaluated with respect to microstructure and density to the requirements of the drawing or applicable specification.

4.14 Records

4.14.1 All records and test results for each hot isostatic pressing treatment shall be maintained for Purchaser surveillance. These records shall include at least the following information:

(a) Purchaser identification of parts or material treated
(b) Part or material alloy designation
(c) Autoclave identification
(d) Pre-cleaning procedures
(e) Loading procedures including fixture materials and part placement
(f) Instrumentation procedures including thermocouple type and placement
(g) Pressure records
(h) Temperature records
(i) Post cleaning procedures
(j) Powder container material and container removal procedures
(k) Pressure media
(l) Metallographic evaluation results
(m) Visual inspection results

4.14.2 Records shall be maintained to provide traceability for each serialized part. Each part shall be traceable to a particular hot isostatic pressure treatment, date, time, autoclave, and location and raw material source.

5. QUALITY ASSURANCE PROVISIONS

5.1 General

5.1.1 All qualifications shall be the responsibility of the hot isostatic pressing vendor. The vendor shall be responsible for all testing and shall sign all necessary forms which certify that qualification in accordance with this specification has been attained.

5.1.2 Procedures for equipment qualifications, if other than those required by this specification, are subject to approval by the Purchaser.

5.1.3 The Purchaser reserves the right to observe any of the qualification tests required by this specification to determine conformance to this specification.

5.2 Autoclave Qualification

5.2.1 Temperature Uniformity. All autoclaves shall be qualified for working zone temperature uniformity prior to use for production hot isostatic pressing. All autoclaves shall be requalified at least every three months after the initial qualification. All autoclaves shall be requalified after any alterations to the equipment which may affect temperature uniformity. Requalification may be on a working load.

5.2.1.1 When approaching thermal equilibrium, per 5.2.2, none of the load temperature readings shall exceed the selected control temperature by more than 25 F (14 C). After thermal equilibrium is reached, the maximum temperature variation of any load test thermocouple shall not deviate from the selected control temperature by more than ± 25 F (± 14 C).

5.2.2 Qualification Procedure. Temperature uniformity tests shall be conducted with the autoclave containing a representative production load of parts or material and at a typical production pressure. The test shall be made using calibrated test thermocouples and a potentiometer type measuring instrument with a minimum sensitivity of 0.02 millivolt. The outputs of the test thermocouples shall be properly corrected as determined by the thermocouple calibrations. A minimum of three test thermocouples or one per each cubic foot (28 dm^3) of working zone, whichever is greater, shall be used for determining the temperature uniformity. When more than three thermocouples are required, the additional thermocouple locations shall be symmetrically distributed within the working zone. The initial qualification shall be performed at the lowest and highest operating temperatures of the autoclave. Requalifications shall be performed at a convenient temperature within the operating range. The temperature of all test and autoclave control thermocouples shall be recorded at five

minute intervals starting immediately after application of power to the autoclave. Temperature measurements shall be continued for at least one half hour after the control thermocouple indicates that thermal equilibrium has been reached so that the recurrent temperature pattern of the autoclave can be determined.

5.3 Temperature Measurement and Control Qualification

5.3.1 Instruments. All instruments used for temperature measurement shall have an indicated temperature accuracy of ± 0.25 percent of the maximum operating temperature over the entire operating temperature range. All instruments used for temperature control shall have an indicated temperature accuracy of ± 0.5 percent of the maximum operating temperature over the entire operating temperature range. The indicated temperature accuracy of each instrument shall be determined in accordance with the equipment manufacturer's recommendations and using a known EMF input of suitable accuracy. After the initial qualification, all instruments shall be requalified at least every 30 days, unless otherwise agreed upon by the vendor and the Purchaser.

5.3.2 Thermocouples. Prior to each use, all thermocouples shall be capable of meeting the temperature - electromotive force requirements of ASTM E230 for special grade wire as determined by suitable test methods and requalification intervals.

5.4 Pressure Indicating Instrument Qualification

5.4.1 All pressure indicating instruments shall be checked in accordance with the equipment manufacturer's recommendations. The equipment's performance shall be within the limits supplied by the equipment manufacturer. After the initial qualification, each instrument shall be requalified at least every six months.

5.5 Process Qualification

5.5.1 Prior to production processing, detailed process procedures and results of test samples shall be submitted to the Purchaser for approval.

5.5.2 Process procedures shall include the following information:

(a) Purchaser identification of parts or material treated
(b) Part and alloy designation
(c) Autoclave identification
(d) Pre-cleaning procedures
(e) Loading procedures including fixture materials and part placement
(f) Instrumentation procedures including thermocouple type and placement

(g) Pressure records

(h) Temperature records

(i) Post cleaning procedures

(j) Powder container material and container removal procedures

(k) Pressure media

(l) Metallographic evaluation procedure

(m) Visual inspection procedure

5.6 Records

5.6.1 All records and test results shall be maintained for Purchaser surveillance. A card shall be affixed to the autoclave and other necessary components after qualification to indicate compliance with this specification. The card shall contain the following minimum information:

(a) Type of equipment

(b) Equipment manufacturer where applicable

(c) Equipment model and serial number where applicable

(d) Equipment location

(e) Statement indicating compliance with this specification

(f) Signature of vendor's qualifying agent.

Appendix F

AUTOCLAVE CYCLES

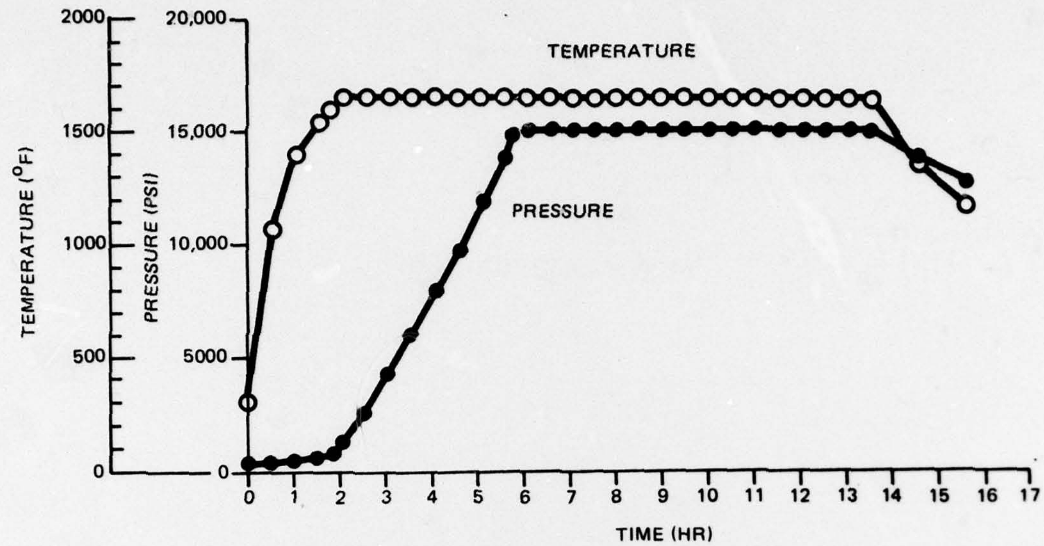

Figure 36 Time-Temperature-Pressure Chart for HIP Run 1 Performed at Kawecki-Berylco

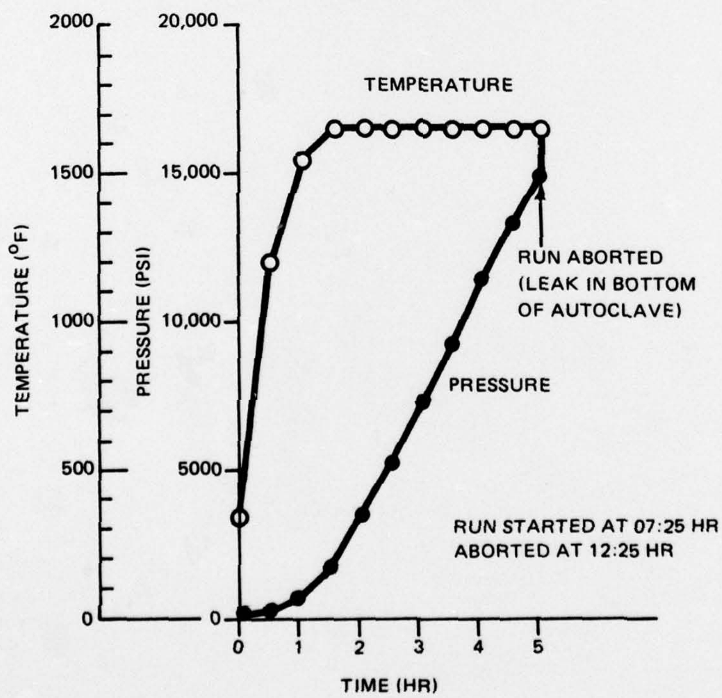

Figure 37 Time-Temperature-Pressure Chart for HIP Run 2 Performed at Kawecki-Berylco

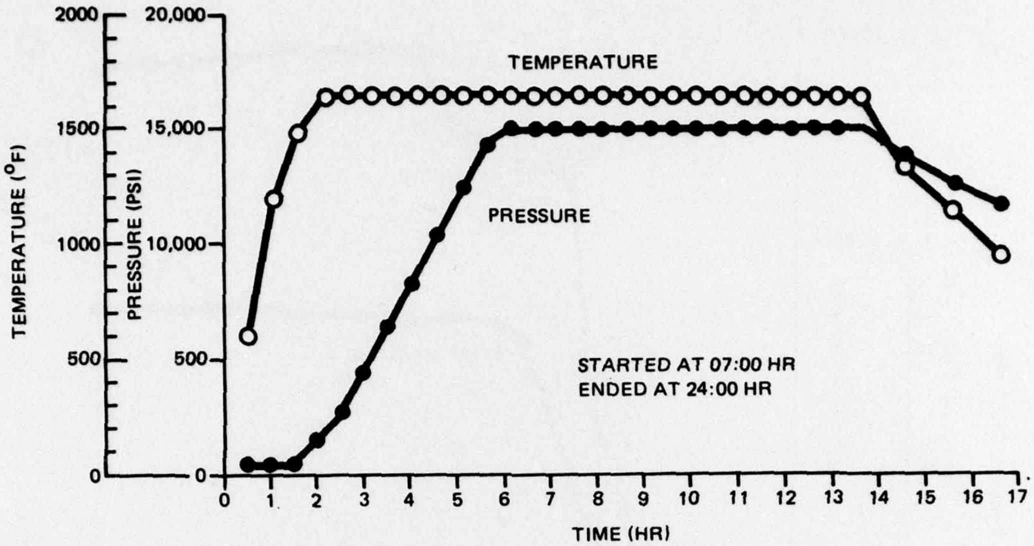

Figure 38 Time-Temperature-Pressure Chart for HIP Run 2 Performed at Kawecki-Berylco

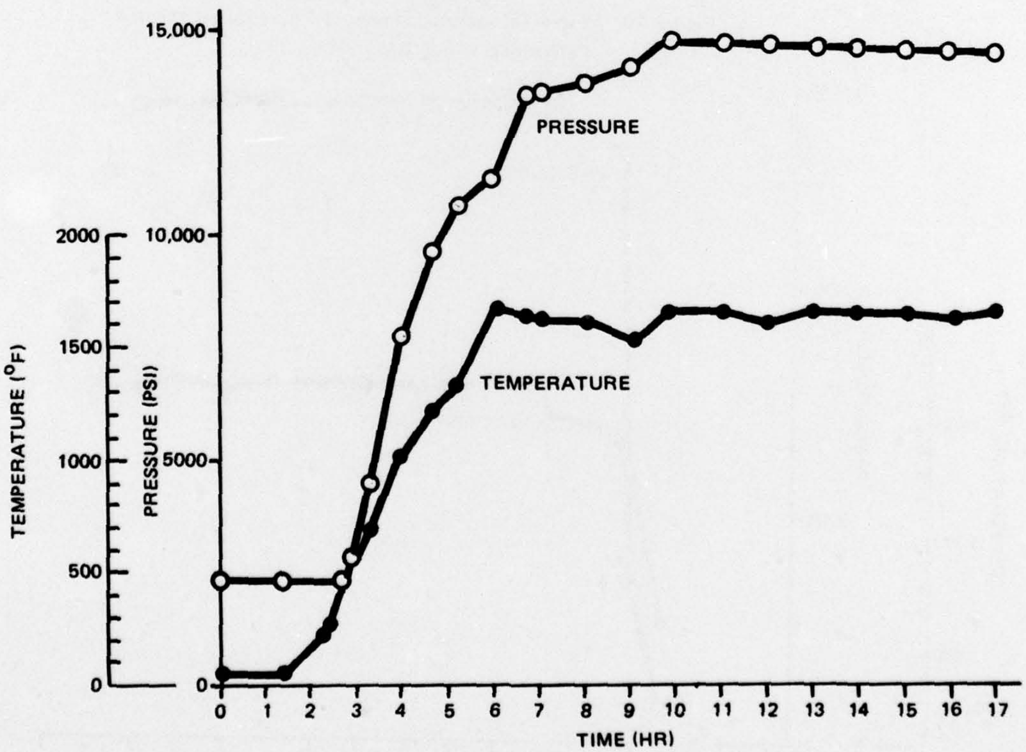

Figure 39 Time-Temperature-Pressure Chart for HIP Run 3 Performed at Battelle

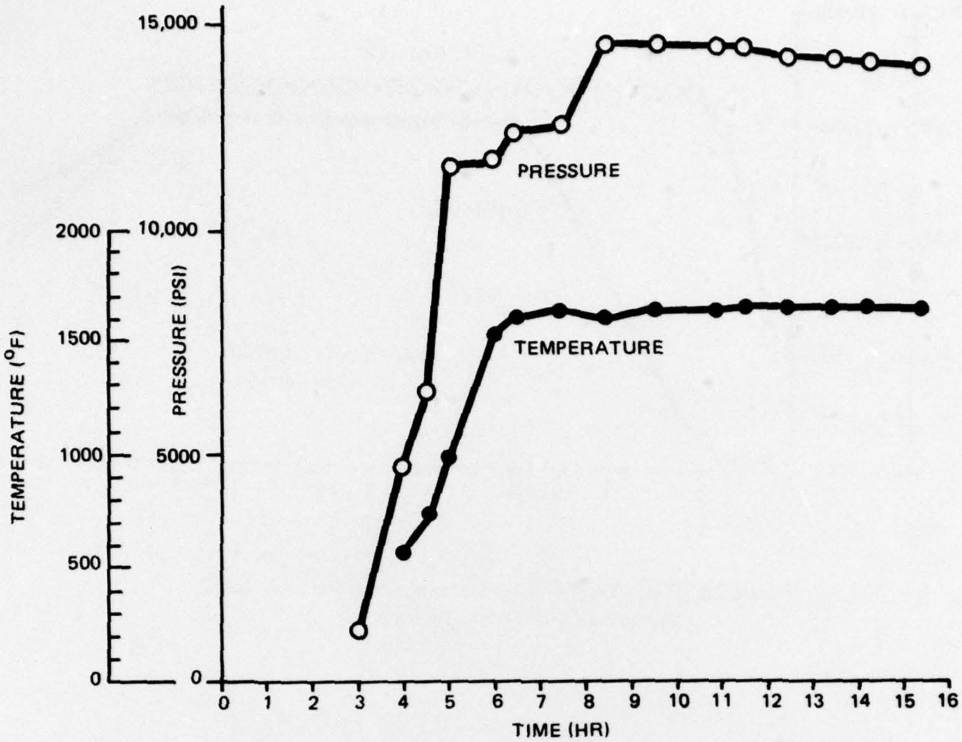

Figure 40 Time-Temperature-Pressure Chart for HIP Run 4 Performed at Battelle

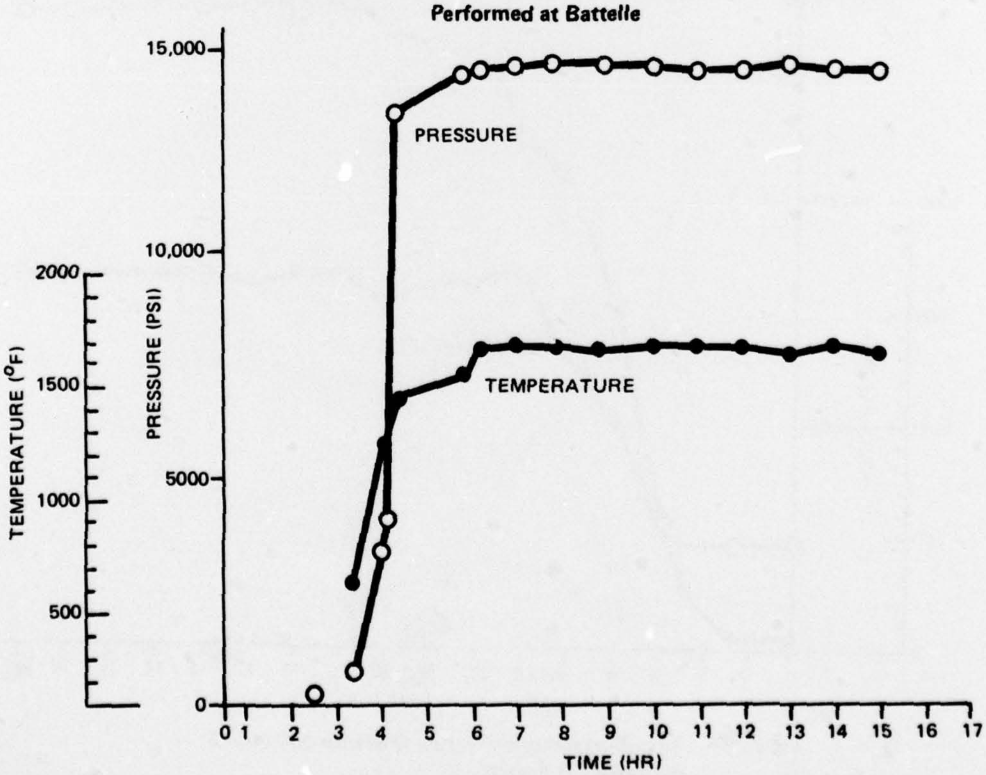

Figure 41 Time-Temperature-Pressure Chart for HIP Run 6 Performed at Battelle

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